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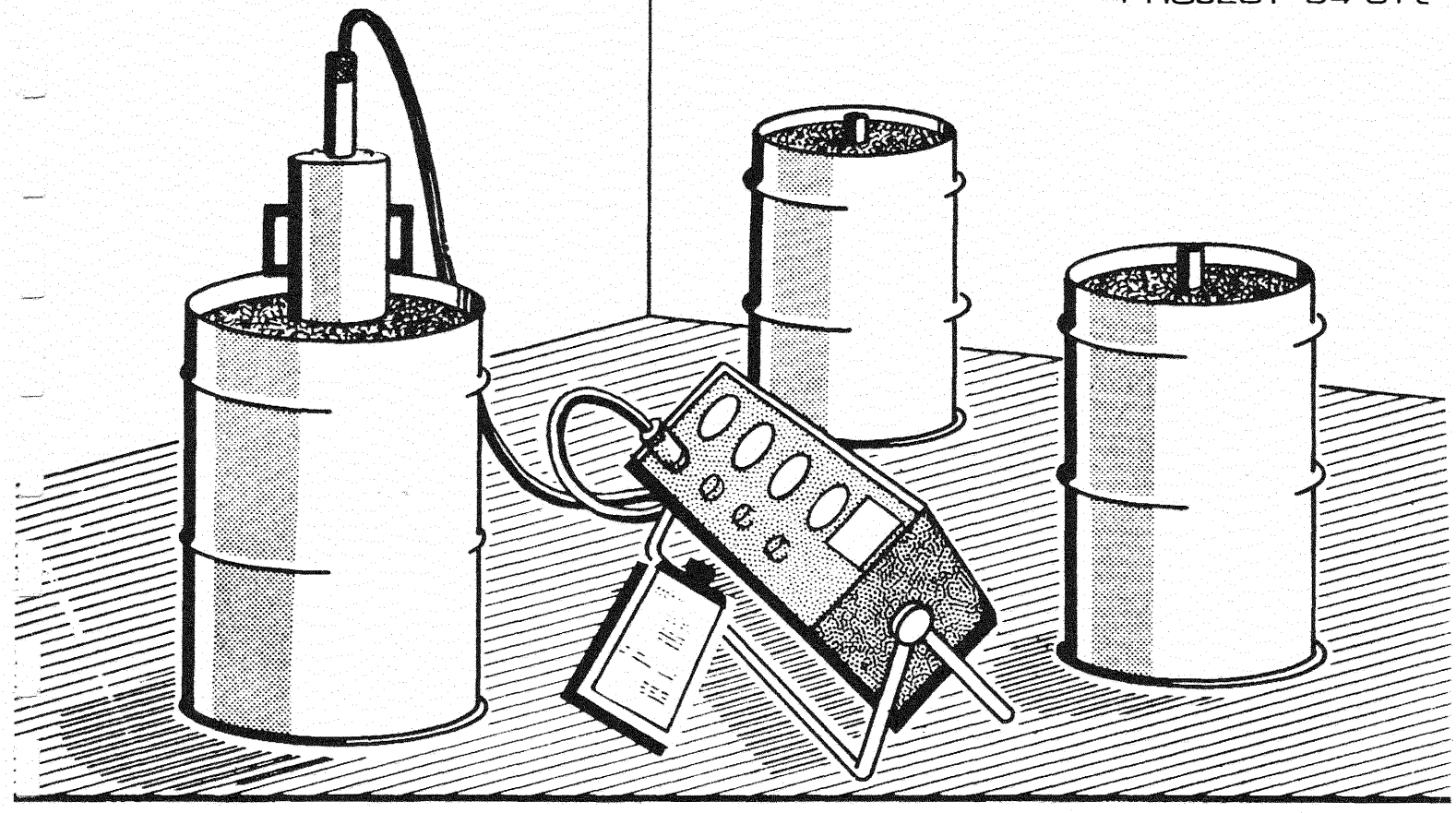

SUBGRADE MOISTURE VARIATIONS

INTERIM REPORT III

A PRELIMINARY STANDARDIZATION AND CALIBRATION PROCEDURE FOR NUCLEAR DEPTH MOISTURE / DENSITY GAGES

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INTERIM REPORT III: A PRELIMINARY STANDARDIZATION AND CALIBRATION PROCEDURE FOR NUCLEAR DEPTH MOISTURE/DENSITY GAGES

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PREFACE

Nuclear equipment for field measurement of soil moisture and density has received wide acceptance from scientists and engineers, but the majority of work accomplished with these instruments has been done with surface type gages. The use of nuclear depth instruments presents some serious problems related to the "standardization" and "calibration" of the equipment. As tentative solutions for some of these problems techniques for the construction and measurement of secondary laboratory standards are presented and a preliminary calibration procedure for both moisture and density gages is proposed.

This report is the third of an interim nature to be submitted by the Subgrade Moisture Variations research project, Oklahoma Research Program Number 64-01-3. Future interim reports will concern preliminary evaluation of field measurements and a more extensive study of the proposed calibration procedure.

Support for this study was provided by the Oklahoma Department of Highways, in cooperation with the Department of Transportation, Federal Highway Administration, Bureau of Public Roads. This support is gratefully acknowledged.

E. W. L.

P. G. M.

LIST OF REPORTS

Interim Report I: "Preliminary Planning," by T. Allan Haliburton, June, 1966, reviews current utilization of nuclear equipment and presents a tentative plan for project operations.

Interim Report II: "Access Tube Installation," by Wayne L. Heiliger and T. Allan Haliburton, January, 1967, describes procedures used to install access tubing for nuclear depth moisture-density equipment beneath highway pavements.

ABSTRACT

A preliminary procedure for standardization/calibration of nuclear depth backscatter moisture and density equipment is described. After a short discussion of theoretical considerations in nuclear measurement of moisture and density, the procedures used to construct secondary soil standards are described, as is the process of data collection. Experimental results are presented and discussed, and recommendations for additional work are given. It is concluded that soil type affects nuclear response in measuring soil moisture and density.

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CHAPTER 1. INTRODUCTION

The physical properties of both compacted and undisturbed soils beneath highway pavements will change over extended periods of time. In some instances the changes may result in improvement of certain soil properties, while in others the changes are considered to be detrimental. It is believed that these changes in subgrade properties from the original, i.e., the conditions that existed at the time a given highway pavement was constructed, are due primarily to changes in moisture content. Such alterations in properties of the subgrade soils may be directly responsible for, or will, at least, contribute largely to the deterioration of the pavement and subsequent high maintenance costs.

There seems to be a general tendency for the moisture content of the subgrade, sub-base, and base materials beneath a highway pavement to increase over a period of time. While some of this increase is due to infiltration of surface water, a large portion is attributed to the migration of moisture in both the liquid and vapor phases from adjacent areas. Regardless of the source, however, the increased moisture content appears to be associated with unsatisfactory pavement performance due to swelling or decreased shear strength of

the subgrade soils. If the extent of this moisture variation beneath existing highway pavements can be determined and all contributing factors evaluated, a correlation with pavement performance will be possible. Ultimately, information of this nature could lead to revisions in present design methods and construction procedures to achieve improved pavement performance.

A cooperative research study to determine (a) the extent to which moisture content of highway subgrade soils is affected by natural environmental factors such as rainfall, temperature, and roadway design characteristics and (b) the relationship between subgrade moisture variations and pavement performance was initiated by the School of Civil Engineering at Oklahoma State University in June, 1964. This research project is sponsored by the Oklahoma Department of Highways and the Bureau of Public Roads.

One of the major investigative problems encountered in the conduct of this research project was concerned with the calibration of nuclear moisture/density gages which were to be used in measuring the field moisture content and density at various depths beneath existing pavements. While the ability of the nuclear or radioactive-type gages to measure moisture content and density of engineering materials is generally acknowledged by scientists and engineers, the calibration of the moisture and density probes presents some serious problems as to the appropriate methods or techniques of calibration. In addition, there is some doubt as to whether experimentally determined calibration curves remain valid as the soil types

or mineralogical constituents of the standards vary (Refs 1, 2).

Faced with these problems, it was considered essential to devote a considerable amount of study to the development of a "standardization" and/or "calibration" procedure for the nuclear probes that would ensure the validity of field measurements made with this type of equipment. The "calibration" of such devices ordinarily refers to the development of a graphical relationship between the pulse rate response of the instrument and either the moisture content or density of the material being studied. "Standardization" normally involves the adjustment of the instrument for such variables as high-voltage drift, changing battery voltage, temperature instability, and drift of the amplifier gain. The need for such adjustments is determined by comparison of a count rate measured on a "standard" with a predetermined value.

This interim report describes the construction of secondary laboratory standards and the development of a tentative calibration procedure. The secondary standards of various constituent materials provide the necessary facilities for standardization of the project nuclear moisture/density equipment and furnish a check on the manufacturer's calibration curves. It is hoped that the tentative calibration technique herein reported will prove to be suitable for all soil types and conditions encountered by the project.

CHAPTER 2. THEORETICAL CONSIDERATIONS

Theoretical considerations are presented in some detail to show that the response of nuclear soil moisture and density measuring devices is a function of elemental composition and mass density of the investigated materials (Ref 3). Although the nuclear interaction processes are complex and highly theoretical it is believed that a semi-empirical solution to calibration is possible which will minimize these material response differences.

Density Determination

The basis for nuclear density determinations is the interaction of gamma photons with the matter through which they are passing. The high energy gamma ray may be thought of as a multitude of particles called photons. These photons are very penetrating but as they pass through matter their number and energy diminish with distance penetrated. Some of these photons are scattered by collision and some are annihilated or destroyed. The denser the mass through which these photons pass, the fewer the photons that will be left after a given distance of penetration. There are three primary modes of interaction and/or absorption of gamma rays. They are designated as the photoelectric effect, the Compton effect, and pair

production. For soil density systems (such as the depth density device) only the photoelectric effect and the Compton effect are significant (Ref 3).

First, in the consideration of the photoelectric effect, an impinging (striking) gamma ray collides with an orbital electron and transfers all of its energy to the electron which is then ejected from the atom. The electron leaves the atom with an energy less than that of the impinging gamma ray. Under certain conditions this ejected electron can create further gamma rays. The photoelectric effect is the predominant mode of gamma absorption for soil materials at energies less than one-half Mev. Generally, it may be stated that, with an increase in the energy of the impinging gamma ray there is an associated decrease in absorption regardless of the type of material; however, different materials absorb at different rates. The probability of a photoelectric interaction is a function of the density of the material, the atomic number and mass of the elements, the percentage of particular elements, and the total number of elements involved (Ref 4).

In considering the Compton effect, the impinging gamma photon collides with an electron and is scattered or deflected, losing a portion of its energy to the electron which is ejected. The electron leaves the atom with a portion of the energy of the impinging gamma photon. The photon continues with less energy but in a different direction. The Compton effect predominates as a way of gamma absorption of energies from 1/2 Mev to 4 Mev. The same relationship between absorption matter and energy of the impinging gamma photon is noted for the

Compton effect as was described for the photoelectric effect (Ref 4).

There are two processes of detecting the amount of interaction which occurs. The scattering process, in which some of the gamma rays are scattered back into the detector, and the direct transmission type where the number of photons which were not scattered are measured. The scattering process complicates the picture in that some of the photons never return and are merely deflected away. Some, however, by several deflections return to the detector to be counted. Each collision will reduce the energy of the photon and a certain average number of collisions are required to redirect a scattered photon back to the detector. Therefore, the backscattered photons that are registered are of very low energy. The measured density is the mass density or the density of the wet solids (Ref 4).

In the depth density device used in this research the measurement procedure makes use of all the photons that reach the detector. Due to deliberate shielding to prevent direct transmitted photons, most are backscattered, see Fig 1. One of the disadvantages to the backscattering process is the large volume of mass involved in backscattering. The net result is that when backscatter photons are counted, the readings are much less sensitive than if direct transmitted photons are the only ones counted. The probability of interaction can be summarized by saying that the primary interaction of gamma rays of high energy with elements of low atomic number is the Compton scattering action and the primary interaction of gamma

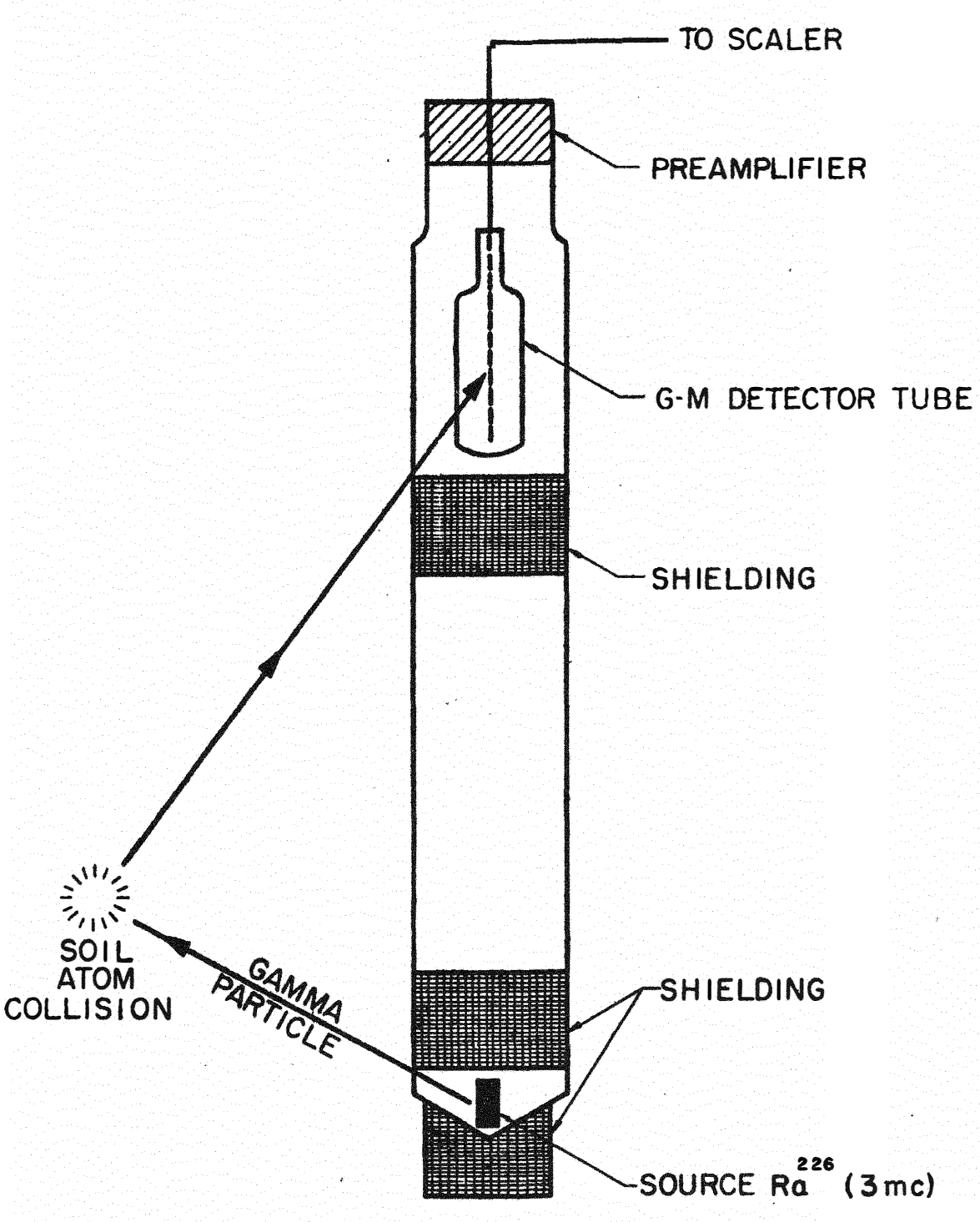

Fig 1. Depth Density Probe

rays of low energy is the photoelectric phenomenon (Ref 3).

Detection of the photons is accomplished by a Geiger-Mueller detection system. A system such as this counts all photons which reach the detector with no discrimination of their energy levels. The distance between source and detector is extremely important since the probability of backscattered photons reaching the detector increases with distance from the source. This requires different calibrations for each particular device. There are so many factors that affect the amount of backscatter that the sensitivity of the unit is poor. However, with proper care and calibration, acceptable results can be obtained.

Moisture Determination

The procedure for measuring soil moisture by the nuclear method consists of placing a source of high energy neutrons near a slow or thermal neutron detector. The number of neutrons that are slowed to thermal energies, i.e., thermal neutrons, in the vicinity of the detector tube is a function of the neutron interactions near this probe. The assumption is made that the response of this device is a function of the hydrogen density near the probe. Secondly, the assumption is made that the hydrogen is present primarily in the water molecule form and that all other hydrogen is insignificant. Therefore, the number of thermal neutrons detected will be a function of the moisture content after a suitable calibration. Theory, as well as experiment, shows that this method will measure hydrogen in the ionic form (Ref 6).

The interaction of neutrons with matter is similar to that of gamma ray interaction. First, there is the elastic collision with atomic nuclei, and second, absorption by the atomic nuclei that results in annihilation of the neutron and subsequently an excited radioactive nucleus. The probability of elastic collision, i.e., elastic particles striking direct and central, is a function of the size and mass of the atomic nuclei. A major consideration is the amount of energy lost in a given collision. This can be explained in terms of basic kinetics. When a particle strikes another particle of equal mass, the impinging particle imparts all of its energy to the struck particle. If the impinging particle has a much smaller mass than that of the nucleus that it strikes, very little of the energy is imparted and the impinging particle is merely deflected in another direction (Refs 6, 7).

If we consider this impinging particle to be a neutron, the only nucleus of similar size and mass would be that of a hydrogen ion, since it consists of a proton which has similar mass to that of a neutron. All other nuclei have significantly larger masses. This is the basis for the assumption that the response of a nuclear moisture probe is a function of the hydrogen density in the vicinity of the probe.

The other mode of interaction, i.e., absorption, is of similar importance. Absorption or capture of the neutron is a function of the capturing element as well as the energy of the neutron. Certain elements such as boron, cadmium, chlorine, and iron will absorb neutrons, particularly the thermal neutrons, to a very high degree. This property is used in neutron detection.

Mathematical equations are available to describe the slowing down process of fast neutrons coupled with diffusion and absorption (Ref 8). This, however, is only of academic interest to the soils engineering application.

In the Troxler depth moisture probe the detection or counting of thermalized neutrons is accomplished by a BF_3 detection system. This detector tube is located as near as possible to a fast neutron source, three millicuries of radium-226-beryllium, situated near the bottom of the probe, see Fig 2. This source is an alpha emitter coupled with a light mass element in which a nuclear interaction occurs that has as a net result the emission of fast neutrons. Some of these emitted fast neutrons are slowed to thermal energies by interaction with hydrogen ions in the vicinity of the probe. A portion of these thermal neutrons are deflected back towards the probe. The BF_3 detector counts these slowed neutrons.

The amount of hydrogen present in soil masses which is not in the water molecule form is minute for most natural formations. Soils with kaolinite clay or high organic content may require separate calibration as they contain significantly higher hydrogen ion concentration. The fact that hydrogen is the only nucleus that will readily thermalize fast neutrons plus the fact that natural soils contain few elements that will capture fast or slow neutrons is most fortuitous. These factors permit soil moisture measurement by nuclear means. The minor discrepancies between theoretical and actual behavior may be accounted for by calibration.

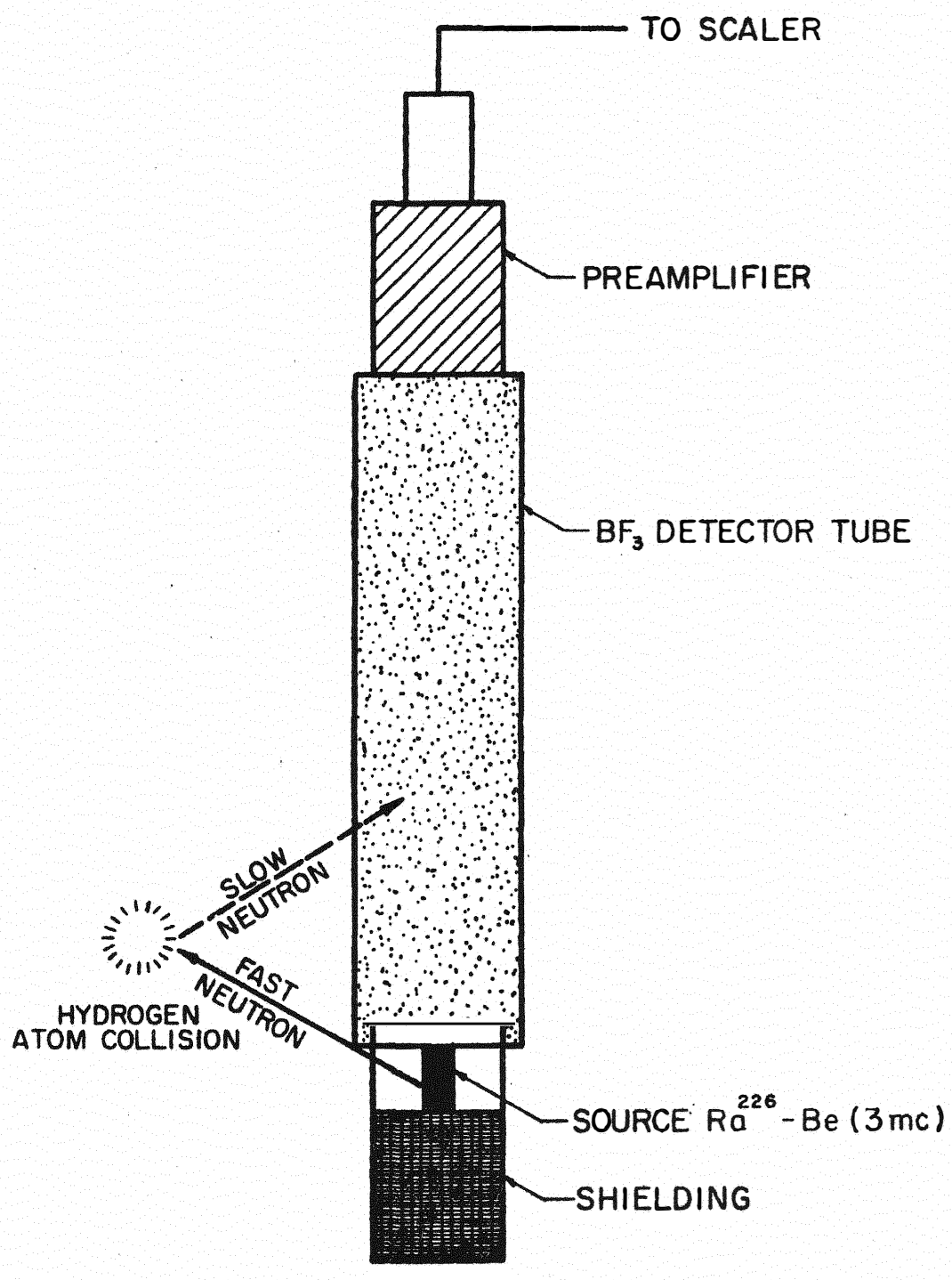

Fig 2. Depth Moisture Probe

CHAPTER 3. EXPERIMENTAL DETAILS

Nuclear Equipment

The nuclear equipment used in this investigation was manufactured by Troxler Electronic Laboratories, Inc., of Raleigh, North Carolina. The counting device was a Troxler Scaler Model 200-B, SN 256. The moisture device was a Troxler SY-SM-1 Depth Moisture Gage, Shield and Standard Model S-5, SN407, with a Depth Moisture Probe Model 104, SN G-19953, using a 3 millicurie Radium-226-Beryllium source, SN N-3-149. The density device was a Troxler ST-DD-2 Depth Density Gage, Shield and Standard Model S-7, SN77, with a Depth Density Probe Model 504, SN 23, using 3 millicurie Radium-226 source, SN-R-3-15. Calibration curves for the moisture and density systems, operational manuals, and radiological safety information were furnished by the manufacturer.

Manufacturer's Standards

The manufacturer's calibration standards were non-soil materials. Portland cement concrete of various unit weights were used for density standards (Ref 1). These standards were constructed using modified 55-gallon oil drums (with approximately one-third of the top removed) as a form or mold. The concrete was placed in these molds around a centrally located access tube and moist cured. After curing the top of the con-

crete was sealed with an epoxy paint to prevent moisture fluctuations. The mass density (actually unit weight) was calculated from weighing the concrete mass and measuring its dimensions. Four barrels of this type were prepared with densities of 108, 114.5, 133.5, and 141 pounds per cubic foot. Three density calibrations of the project equipment were made with these standards during the period January 5 to November 19, 1965.

For moisture standards the manufacturer used distilled water-cadmium chloride solutions of varying concentration (Ref 1). Cadmium has a high probability of capture for slow neutrons, i.e., the most probable nuclear reaction is for the cadmium to take the neutron into its nucleus. With an increase of cadmium present, fewer neutrons will be elastically scattered and returned to the detector. The water solute provided a means of thermalizing fast neutrons. The various concentrations used were correlated to equivalent moisture contents by field testing typical North Carolina soils. This provided a simple factory standard. The solutions were kept in 30-gallon waste cans with a provision for placement of a central access tube. By changing the size of the access tubes, different diameter probes can be calibrated with a single set of standards. Moisture calibrations with these standards were performed on December 10, 1964, and November 19, 1965.

Project Standards

Because of the weight and storage problems involved in constructing secondary standards it was considered desirable

to attempt the development of standards that could be used for both density and moisture content calibration. A considerable amount of work was one on the use of "polyethylene concrete," various percentages of polyethylene pellets in a sand-portland cement mortar matrix, for this purpose. However, this idea, although quite feasible, presented some difficult problems and was abandoned due to certain time schedule limitations of the project.

Materials

Three locally available types of aggregate were selected for use in the secondary standards. These aggregates were a crushed limestone, a river gravel, and a lightweight concrete aggregate--expanded shale. Since calcareous and silicious sedimentary deposits predominate throughout most of Oklahoma, it was considered that these three materials while serving as standard media might also provide a basis for evaluating the effects of mineral type variance found in the different soils encountered during the field testing phase of the project.

The apparent specific gravity of the respective aggregates was as follows:

Crushed Limestone	2.68
River gravel	2.64
Expanded shale	2.71

The aggregate gradation used for the standards was:

Crushed limestone ... -1/2" and + No. 4 sieve.
River gravel .... -No. 4 and + No. 10 sieve.
Expanded shale .... -1/2" and + No. 4 sieve.

A single size gradation was used in all cases in order to achieve a more uniform density of the materials in place in the standard barrels. About two tons of the commercial aggregates were screened to get these particular sizes in the quantity desired.

In addition to the aggregate standards, three standard barrels were prepared using Permian red clay from a source located on the OSU campus. This clay material had a specific gravity of 2.72, a liquid limit of 40.6 and a plastic limit of 15.0 (Ref 10).

Aggregate Standards

The first standard prepared with the crushed limestone utilized a 30-gallon galvanized waste can as the container. Vibration of the aggregate during placement caused distortion in the light gage metal of the can and changing of its volume. Because of this, cut-down 55-gallon oil drums were used for all subsequent standards constructed.

Sections of seamless aluminum irrigation tubing 25 inches long were used for the access tubes which were centrally located in the standard containers, see Fig 3. These access tubes were plugged at the bottom end with a lead disc and O-ring arrangement, see Fig 4, and then the plugged ends were sealed with an epoxy paint.

Drainage was provided at the bottom of the standard containers. For the 55-gallon oil drums, drainage was facilitated by using the top of the barrel as the bottom of the standard container. Standard galvanized pipe fittings screwed into

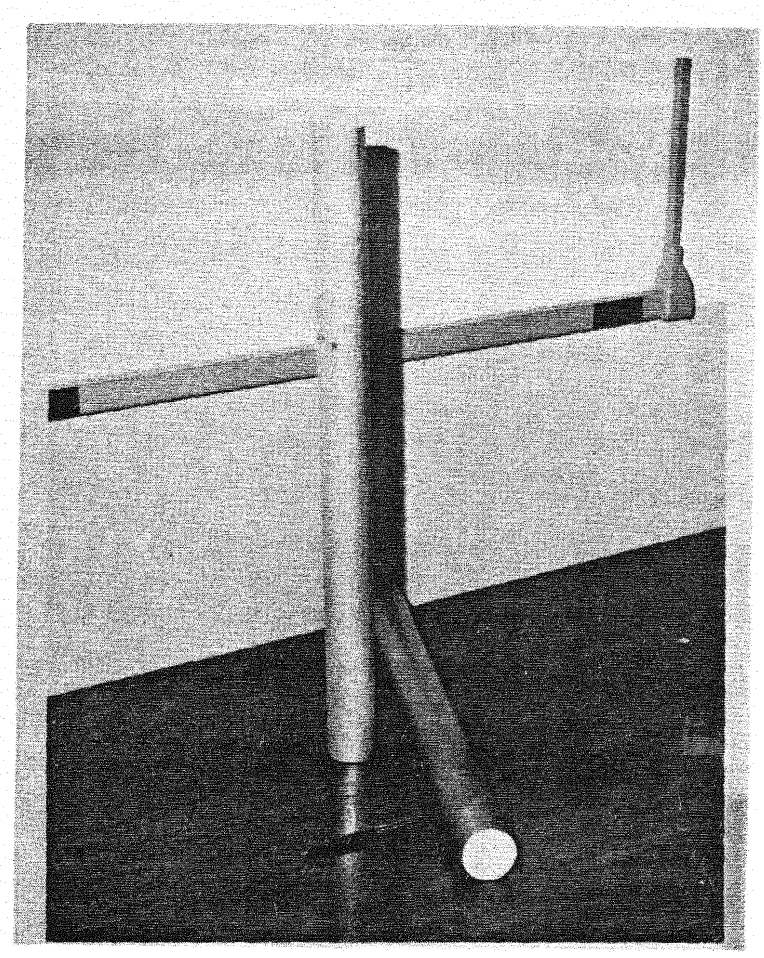

Fig 3. Aluminum Access Tubes

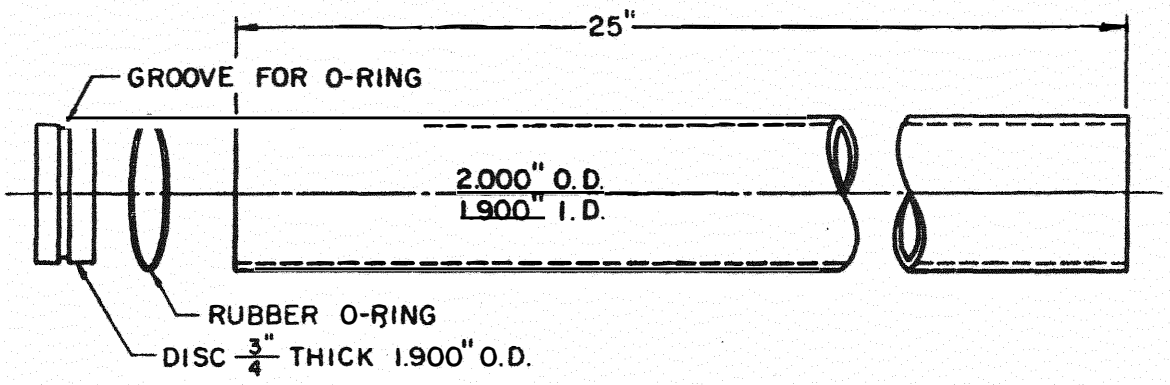

Fig 4. Sealed End of Aluminum Access Tube

the small bung provided the necessary drainage system. A line or mark was scribed and painted just below the tops of the standard barrels and served as a fill-line or upper limit for the aggregate samples. The volumes of the containers were calculated from weight of water determinations. The average diameter of these cut-down drums was 23 inches and they had an average volume of 5 cubic feet. The 30 gallon galvanized waste can had an average diameter of 18 inches and a volume of 2.6 cubic feet.

The construction procedure for all the aggregate standards was the same. One size gradations were obtained by sieving the aggregate with a Gilson sieve shaker. Following this, the aggregates were thoroughly washed to remove all fine material. The aggregates were then oven-dried to constant weight and placed into the container while still hot. Fig 5 shows an empty standard barrel with the central access tube in position prior to the placement of the aggregate. Notice the screen mesh placed over the drain outlet. Placement was done in three lifts using a small concrete vibrator to achieve maximum density. Fig 6 shows a partially filled barrel and Fig 7 shows a filled barrel with the vibrator used for densification of the aggregate materials.

After placement the surface of the final layer of material was carefully leveled and the filled containers were covered with plastic sheeting, sealed, and allowed to cool to room temperature. The standards were then weighed on a Fairbanks-Morse platform scale, sensitive to one-half pound, and dry unit weights were calculated. After weighing the standards

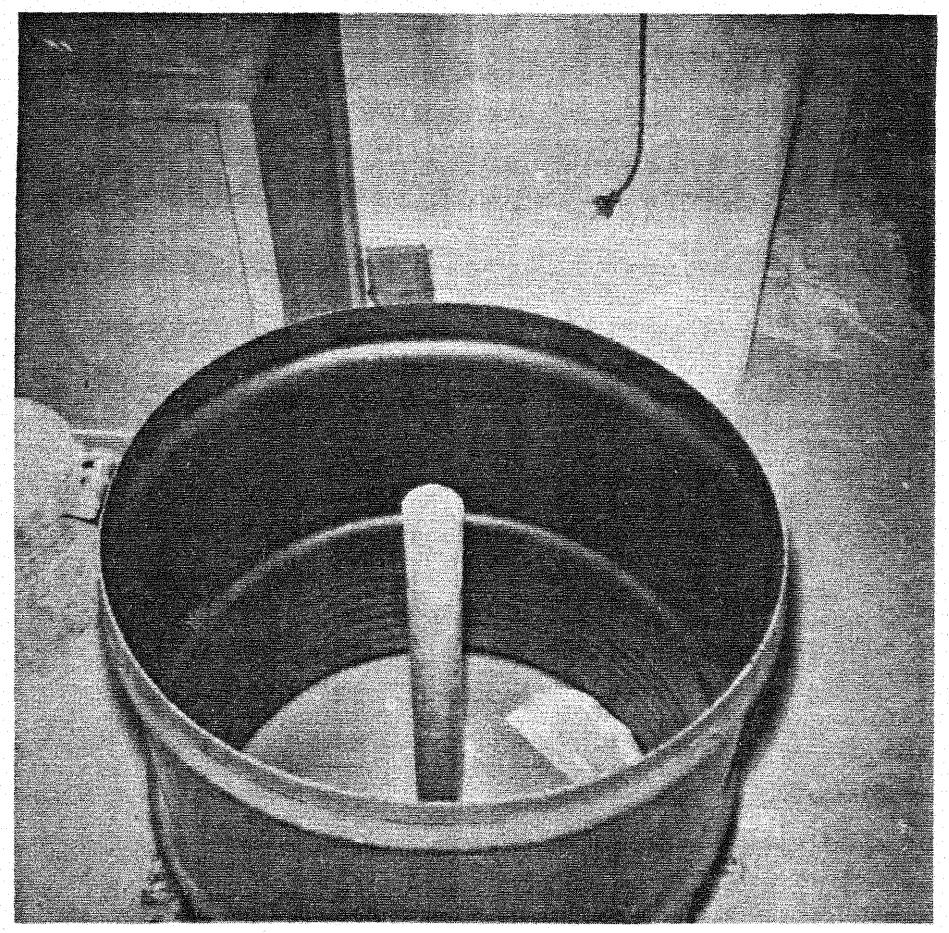

Fig 5. Empty Standard Barrel With Central Access Tube in Position

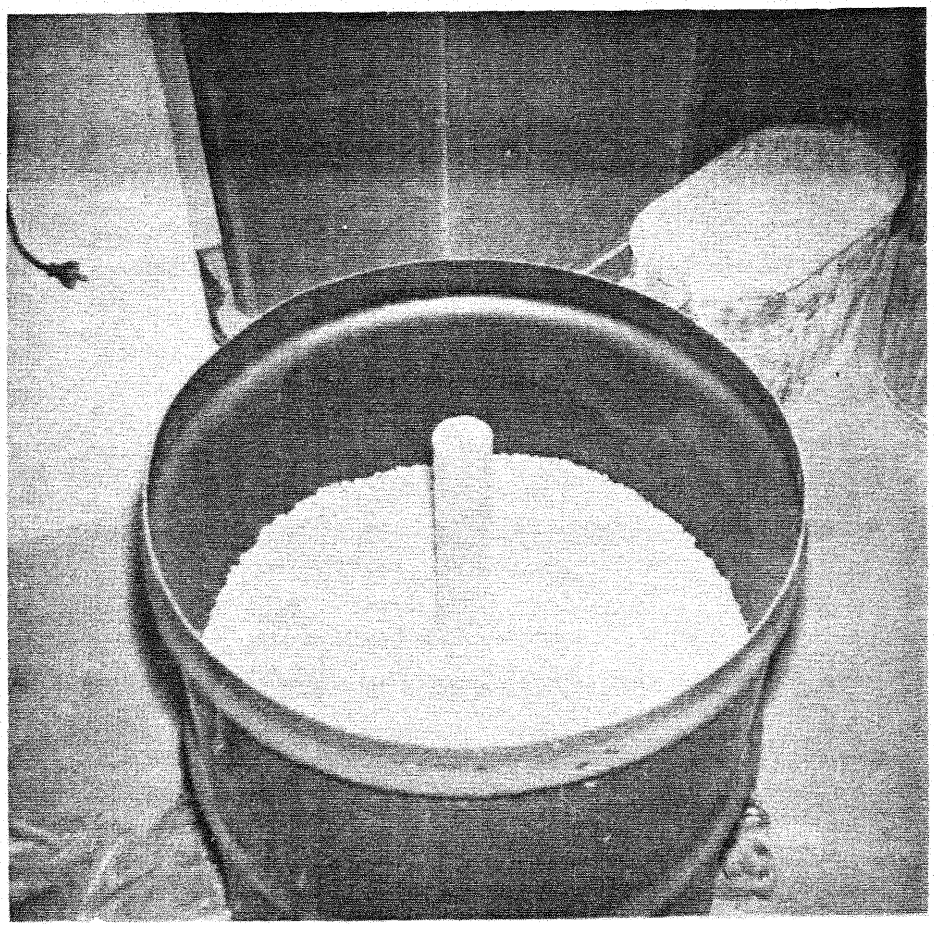

Fig 6. Partially Filled Barrel Standard-Crushed Limestone

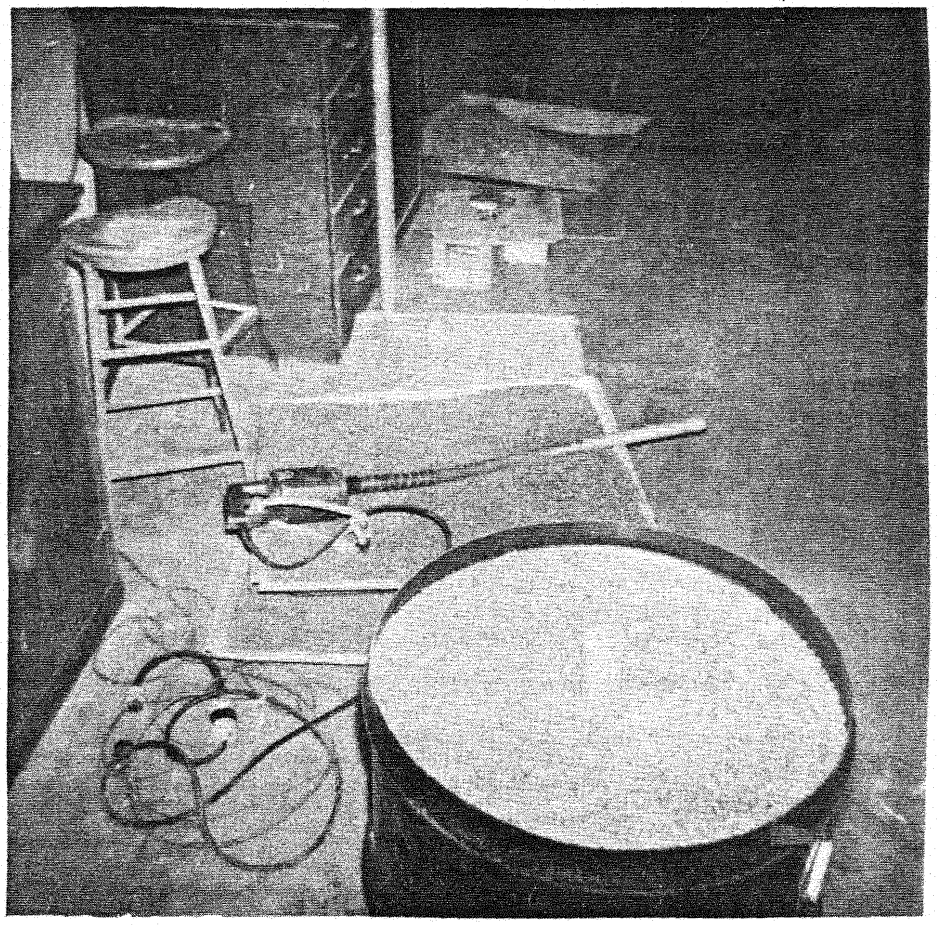

Fig 7. Filled Barrel Standard-River Gravel-Drying Pans and Vibrator in Background

were "read" with both the moisture and density devices. Fig 8 shows the density probe in the shield and standard as reference readings are being made. Fig 9 shows the density probe in position for readings on a standard barrel immediately after weighing and in Fig 10 readings are being made on a standard barrel with the moisture probe. In the left foreground of Fig 10 can be seen some of the plastic sheeting used as a moisture barrier for the standards.

Following the initial "dry" readings with the nuclear probes the aggregate standards were flooded with hot (200°F) water. The water was introduced at a slow rate through the bottom drain connections of the container until it completely covered the aggregate. The combination of hot water and slow filling from the bottom helped to minimize dissolved as well as entrapped air in the flooded standards. After flooding, the aggregates were allowed to soak for at least seven days before the standards were weighed and the respective wet unit weight and amount of water on a volume basis were calculated.

At this stage of the preparation rusting of the barrels at the water level was noticed in the first few barrels that were constructed. This problem was alleviated by the use of an epoxy paint on the inner surface of the barrels.

The expanded shale standard required three weeks of soaking before an "assumed" state of saturation was reached. Preliminary soaking tests on samples of this material revealed that after soaking periods of up to six weeks the expanded aggregate was still absorbing small amounts of water. Since the major portion of absorption occurred during the first three

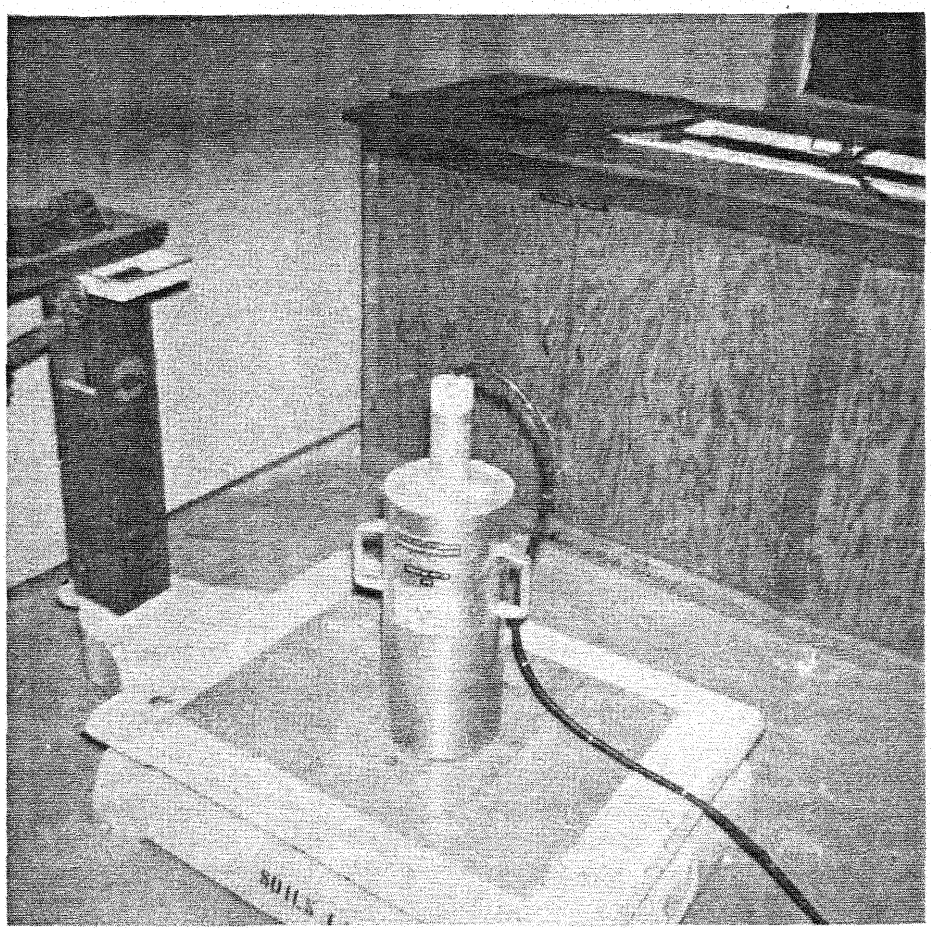

Fig 8. Density Probe in Shield and Standard

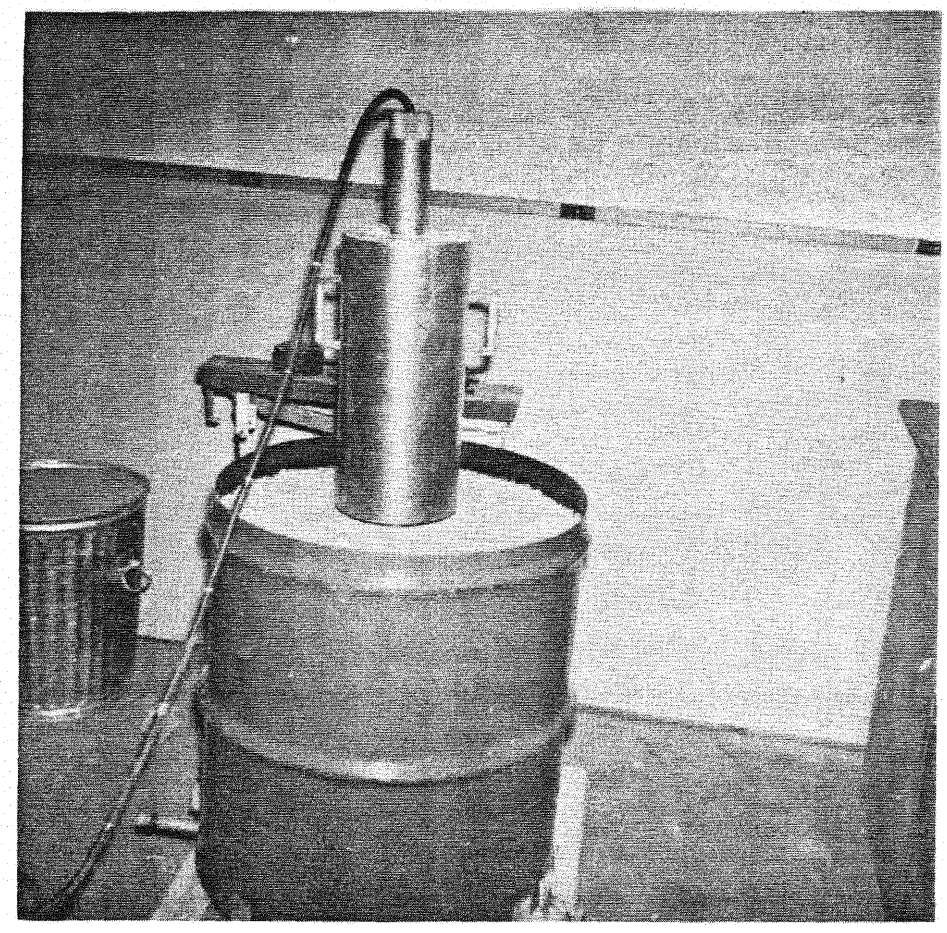

Fig 9. Density Probe in Position for Readings on Standard Barrel

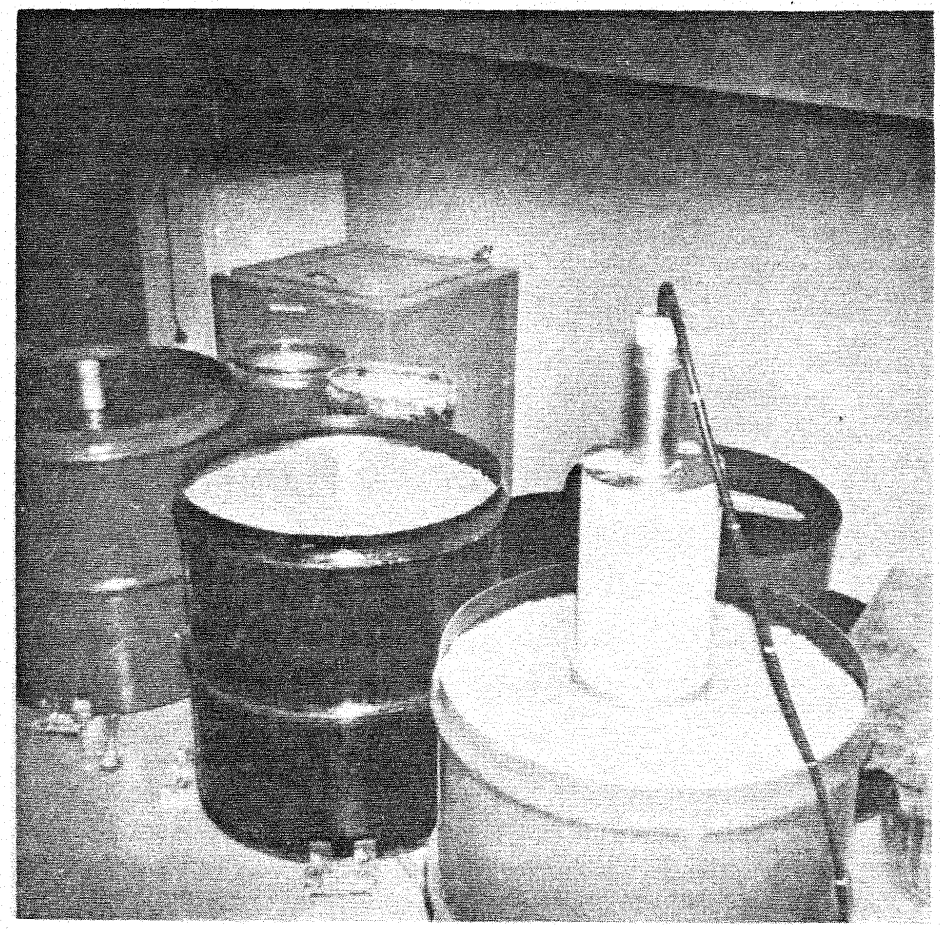

Fig 10. Moisture Probe in Position for Readings on Standard Barrel

weeks this was the soaking period adopted for the barrel standard. A slight amount of settlement was noticed in the expanded shale standard during the soaking period although none occurred in the others. This required a recalculation of the volume occupied by the aggregate.

After soaking the necessary time, the proper probe position in the access tube was determined and moisture and density readings were taken. As soon as the saturated readings were taken, the standards were allowed to drain. Plastic covers were placed over the tops of the barrels and the air space between the cover and aggregate was filled with additional plastic sheeting. This served to prevent evaporation from the surface of the aggregate. The standards were drained for three days and then reweighed to determine the drained unit weights. The proper placement of the probes was determined and readings taken on the standards in the drained condition. Each aggregate type furnished three points on a calibration curve and an additional set of values was determined from the 30-gallon waste can standard.

Clay Standards

After consideration of the data obtained from the aggregate standards and recommendations found in the literature to check the calibration of the nuclear probes on the materials with which the instruments are to be used (Refs 1, 3), three additional barrel standards were prepared using Permian red clay. This material was thought to be the major soil type that will be encountered.

The clay was first air-dried and then pulverized, using a Los Angeles abrasion machine as a ball mill, until it passed a U.S. No. 30 mesh sieve. Three moisture contents were selected for the barrel standards. Allowing for the initial moisture content of the material the necessary amount of mixing water per 50 pounds of material was determined. This water was added to the pulverized clay through a fine spray nozzle and 50 pound batches were mixed by hand. After the water was incorporated the moistened clay was sealed in 30 gallon waste cans for several days until an assumed moisture equilibrium condition existed. This was determined by visual examination of the texture of the material as well as by the feel of the mixture in the hand. Since each barrel standard required three 30-gallon cans of loose mixture, the material in each compacted lift was obtained by alternately removing equal amounts from each of the cans.

Compaction of the material in the barrels was achieved using a Thor (size 661) pneumatic backfill tamper weighing approximately 30 pounds. The foot on this tamper was worn excessively and a circular steel plate the same diameter as the foot and 3/8 inch thick was bolted to the bottom of the foot to allow compaction at the periphery of the clay material, adjacent to the barrel wall. A regulator in the air line was used to provide a constant pressure of 50 psi to the tamper. To prevent displacement of the bottom during compaction a circular plywood disc was placed under the barrel.

The material was compacted in lifts of approximately two inches (compacted thickness). A standard compactive effort

for each lift was achieved by maintaining a constant period or time of compacting with the pneumatic tamper using a stop-watch. With this method the compactive effort could be varied by lengthening or shortening the compacting period.

After the barrel was filled to the predetermined volume mark, the surface of the material was carefully leveled and the weight determined with the platform scale. Due to the size and weight of the stands a cross shaped steel platform and chain sling arrangement was developed for use with an overhead crane to lift and manuever the completed barrel standards, see Fig 11. After weighing, the wet density of the material was calculated.

To install the access tubes in these compacted clay standards a two inch O.D. steel tube was slowly pushed into the center of a compacted barrel. This steel tube had a 1/16 inch wall thickness and was sharpened on the inside at one end to minimize material disturbance as it was forced into the clay, see Fig 12. Forcing of this cutting tube into the compacted clay was accomplished using a Tinius Olsen 200,000 pound Universal Testing machine. After the steel tube was withdrawn, an aluminum access tube, sealed on the bottom end, was inserted in the cored hole. The core sample obtained from the steel tube was used to determine the percent water content on a weight basis. Using the wet density and water content (weight basis) the water content per unit volume was calculated.

Following the installation of the aluminum access tube, the proper positions for the probes were determined and moisture and density readings made. Plastic sheeting was used as

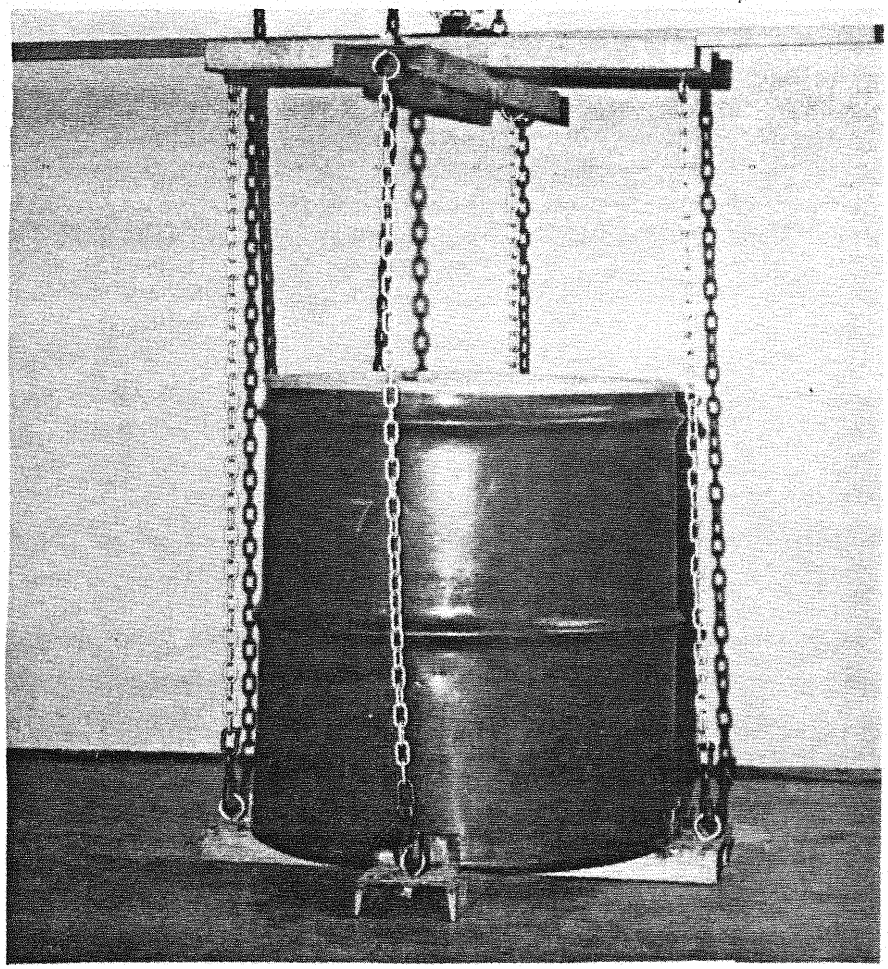

Fig 11. Steel Platform and Chain Sling

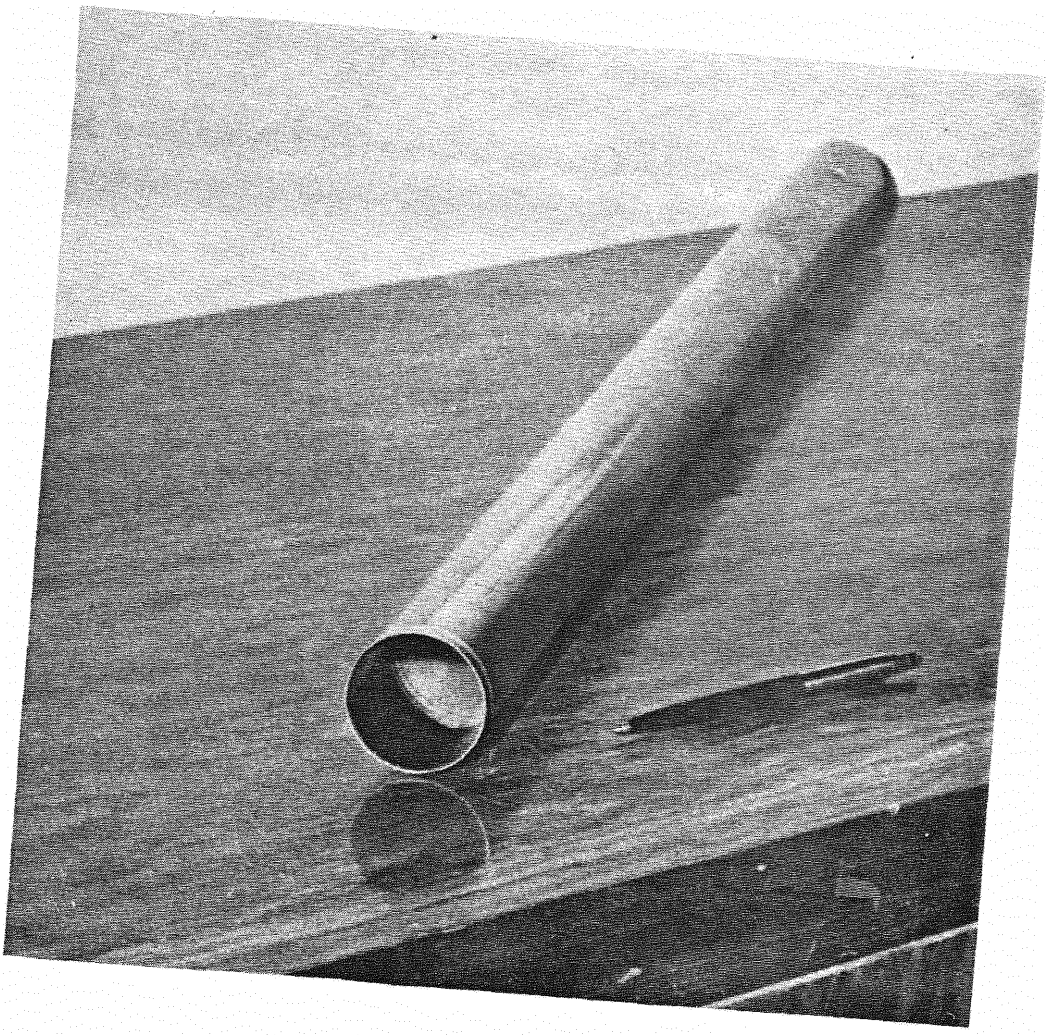

Fig 12. Sharpened Steel Cutting Tube

in the aggregate standards to seal the clay barrel standards and prevent surface evaporation.

Probe Location

The proper positioning of the probe in the access tubes of the barrel standards was determined by taking readings at one-inch intervals from the bottom. The data (in graphical form) were used to select a location for probe placement. A section where little change in reading occurred with probe location change was used. For the density probe this location was near the bottom of the access tube. The very bottom position was not desirable due to backscattering from the container bottom and platform. The location of the moisture probe was near mid-height of the standard. The sphere of influence of a moisture probe is a function of moisture present and the center of this sphere is a function of the geometry of the source and detector. For these reasons the proper probe location was determined for each individual standard in each of the respective conditions, i.e., the dry, soaked, and drained conditions.

Calibration curves were plotted as a soil mass property versus percent of standard count. The percent of standard count method is less subject to error than the absolute count method since it compensates for electronic instabilities. In the OSU laboratory procedure twenty (ten counts before and ten counts after) standard counts were taken whenever soil counts were made. Standard counts were taken in the shield unit utilizing sources imbedded in the standard cylinder. The operating voltage was determined for each set of instruments

using the "plateau" principle. This is a voltage range where the effect of voltage change on pulse output is at a minimum. The scaler unit was connected to an outlet during all laboratory measurements to eliminate changes in applied voltage due to discharge of the battery.

CHAPTER IV

DATA PRESENTATION AND DISCUSSION

Table 1 shows a summary of the depth density probe readings obtained on the various OSU constructed standards and the Troxler concrete standards. The standard material and its condition, i.e., dry, drained, or saturated, is indicated in the first column of this table. The true mass density of these materials in pounds per cubic foot was obtained from their weight-volume relationships. Values of mass density shown in the extreme right hand column were calculated from the probe reading using the linear equation for the recommended calibration curve. The development of this curve is explained in the following discussion of the density data. Readings obtained in air and water are shown for purposes of comparison.

The data presented in Table 2 is a summary of the depth moisture probe readings for the various OSU standards and the Troxler cadmium chloride solutions. The computed water contents shown for the granular materials in the saturated condition and the Permian clay standards are based on the indicated probe reading and were determined using a linear equation for a portion of the recommended moisture calibration curve developed from the data.

Density

According to theory the relationship between mass density

TABLE 1

DATA SUMMARY - DENSITY

Type of Material	True Mass Density (pcf)	Probe Reading (% std)	Computed Mass Density from Curve (pcf)
Concrete*	108	29.81	107.1
Concrete*	114.5	27.28	113.4
Concrete*	133.5	20.50	135.7
Concrete*	141	18.21	144.7
Concrete**	108	30.99	104.2
Concrete**	114.5	28.33	111.0
Concrete**	133.5	21.34	137.6
Concrete**	141	19.21	140.6
Sat. Limestone***	134.3	21.56	131.8
Drained Limestone***	110.5	29.44	108.1
Dry River Gravel	104.5	31.64	102.6
Sat. River Gravel	126.4	23.46	125.4
Drained River Gravel	107.5	30.22	106.1
Dry Limestone	95.6	32.90	99.6
Sat. Limestone	123.9	22.60	128.3
Drained Limestone	98.1	31.74	102.4
Dry Expanded Shale	56.3	51.29	--
Sat. Expanded Shale	87.3	38.78	86.6
Drained Expanded Shale	64.6	48.02	--
PRC****	116.3	22.64	--
PRC****	122.7	24.18	123.1
PRC****	110.3	27.47	113.4
Air	-0-	9.04	--
Water	62.4	58.95	--

* Troxler Standard April 9, 1965

** Troxler Standard November 19, 1965

*** Waste Can Standard

****Permian Red Clay

TABLE 2

DATA SUMMARY - MOISTURE

Type of Material	Water (pcf)	Probe Reading (% std)	Computed Water Content from Curve (pcf)
Troxler*	18.91	61.46	--
Troxler*	15.58	49.68	--
Troxler*	10.17	35.36	--
Troxler*	6.61	24.36	--
Troxler*	62.4	147.33	--
Troxler**	18.91	62.43	--
Troxler**	15.58	51.46	--
Troxler	10.17	38.17	--
Troxler**	6.61	27.44	--
Troxler**	62.4	153.86	--
Dry Limestone***	-0-	0.9	--
Sat. Limestone***	26.9	107.6	26.7
Drained Limestone***	3.1	3.3	--
Dry River Gravel	-0-	1.16	--
Sat. River Gravel	21.9	90.24	22.5
Drained River Gravel	3.0	5.11	--
Dry Limestone	-0-	1.65	--
Sat. Limestone	28.3	114.82	28.4
Drained Limestone	2.5	6.32	--
Dry Expanded Shale	-0-	0.60	--
Sat. Expanded Shale	31.0	88.72	22.1
Drained Expanded Shale	8.4	12.68	--
PRC****	11.8	56.41	14.3
PRC****	19.3	74.25	18.6
PRC****	12.2	48.74	12.4
Air	-0-	0.24	--
Water	62.4	158.04	--

* Cadmium Chloride Solutions December 10, 1964

** Cadmium Chloride Solutions November 19, 1965

*** Waste Can Standard

****Permian Red Clay

Density

According to theory the relationship between mass density and percent standard count is approximately linear on a semi-logarithmic plot for values of normal soil mass densities. At least, the relationship is linear for this type of plot for densities above about 90 pcf (Ref 1). Density data obtained in this study on each of the standards has been plotted with percent standard counts as the ordinate on a log scale and wet density in pounds per cubic foot as the abscissa on an arithmetic scale. A least-squares regression analysis was made on each set of data and an equation for the linear portion of the curve was determined. A sample of the computation sheet for the least-squares regression line analysis on the Permian red clay standards is shown in Fig A-1 in the Appendix.

Figures 13 and 14 show the data obtained from readings on the Troxler concrete standards. Figure 13 is derived from data supplied with the depth density probe and Fig 14 is from data taken several months later on the same set of standards. The two curves have essentially the same slope but the later data curve is shifted slightly upward. This is due to a difference in the number of reference standard counts made when the data was obtained, the April 9, 1965 data being based on a smaller number of reference standard counts.

Figure 15 is a plot of the data obtained from the first OSU standard, i.e., a 30-gallon waste can filled with crushed limestone aggregate. Only two density measurements were available from this standard. The curve for the river gravel standard is shown in Fig 16. Interestingly, the curve equation for these two materials, the limestone and the river gravel,

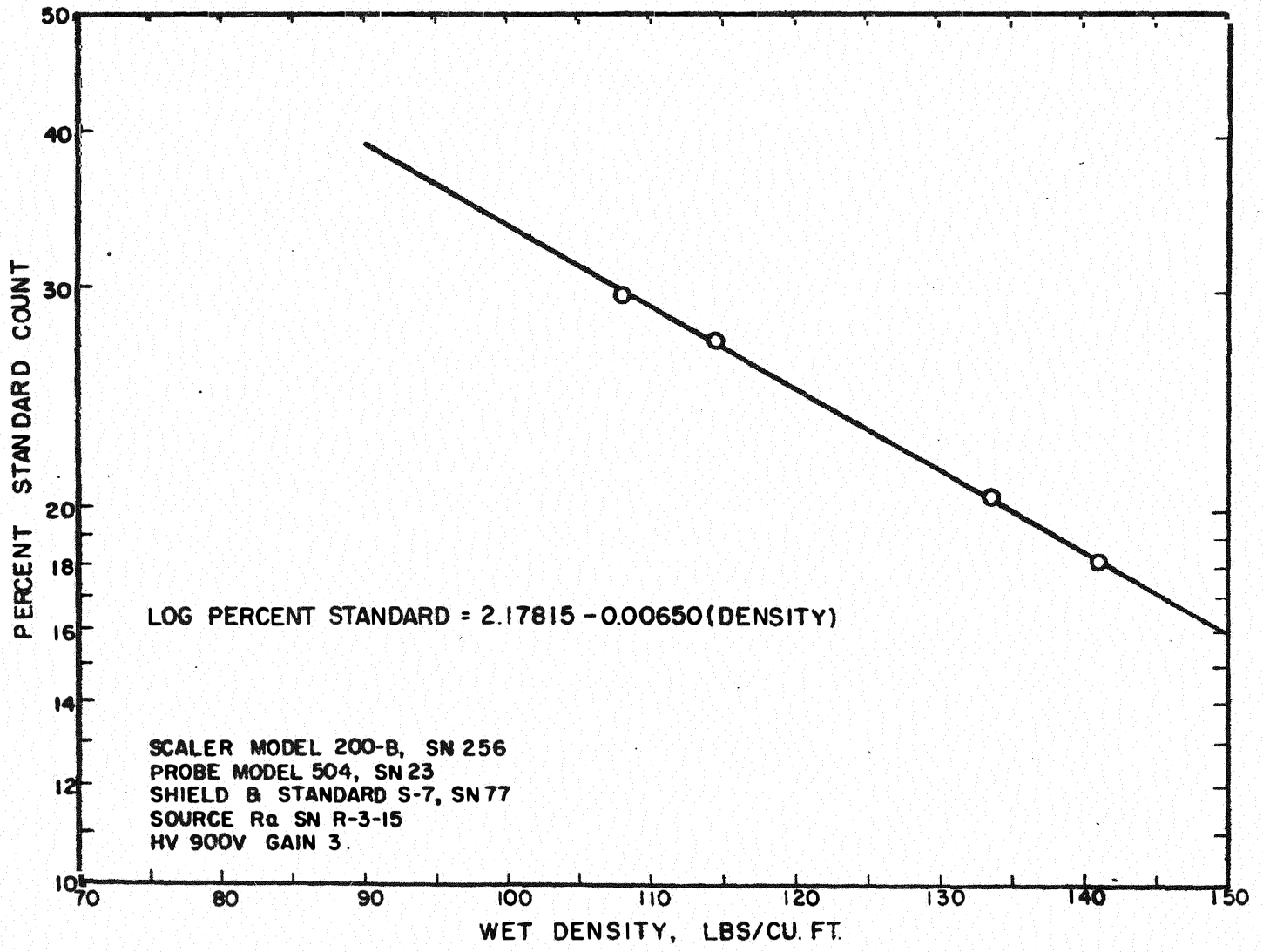

Fig 13. Density Calibration Curve-Troxler Concrete, April 9, 1965

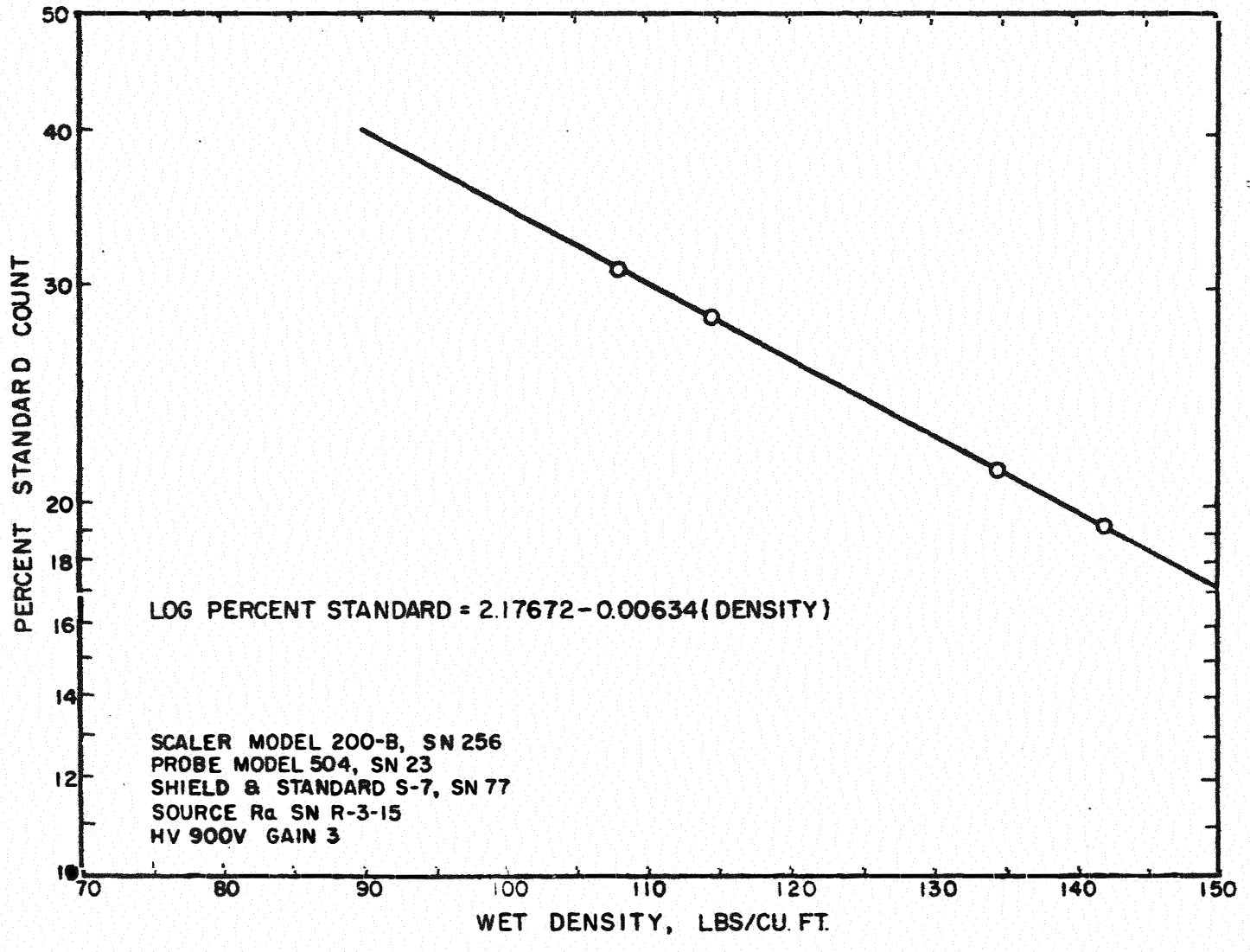

Fig 14. Density Calibration Curve-Troxler Concrete, November 19, 1965

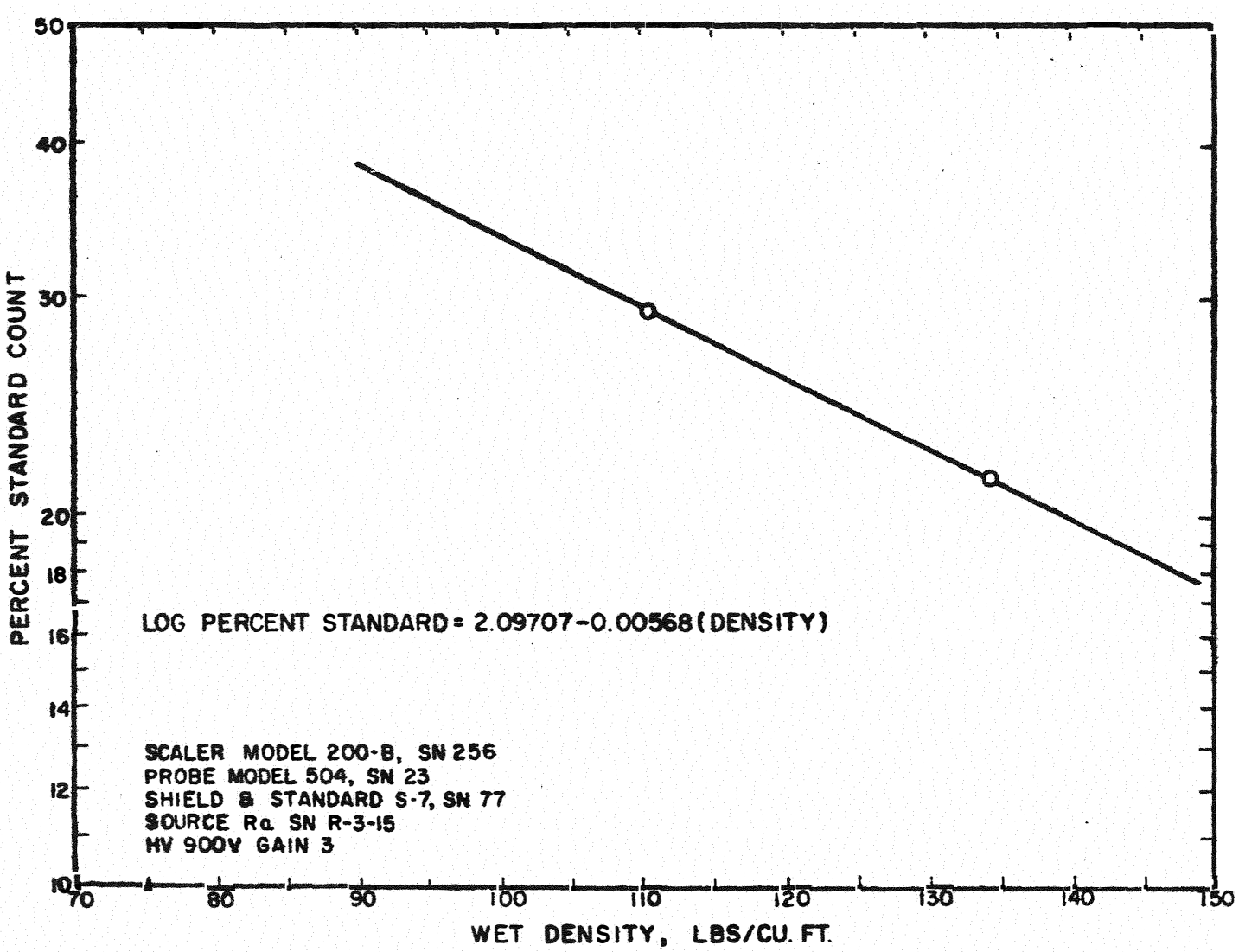

Fig 15. Density Calibration Curve-Limestone Waste Can

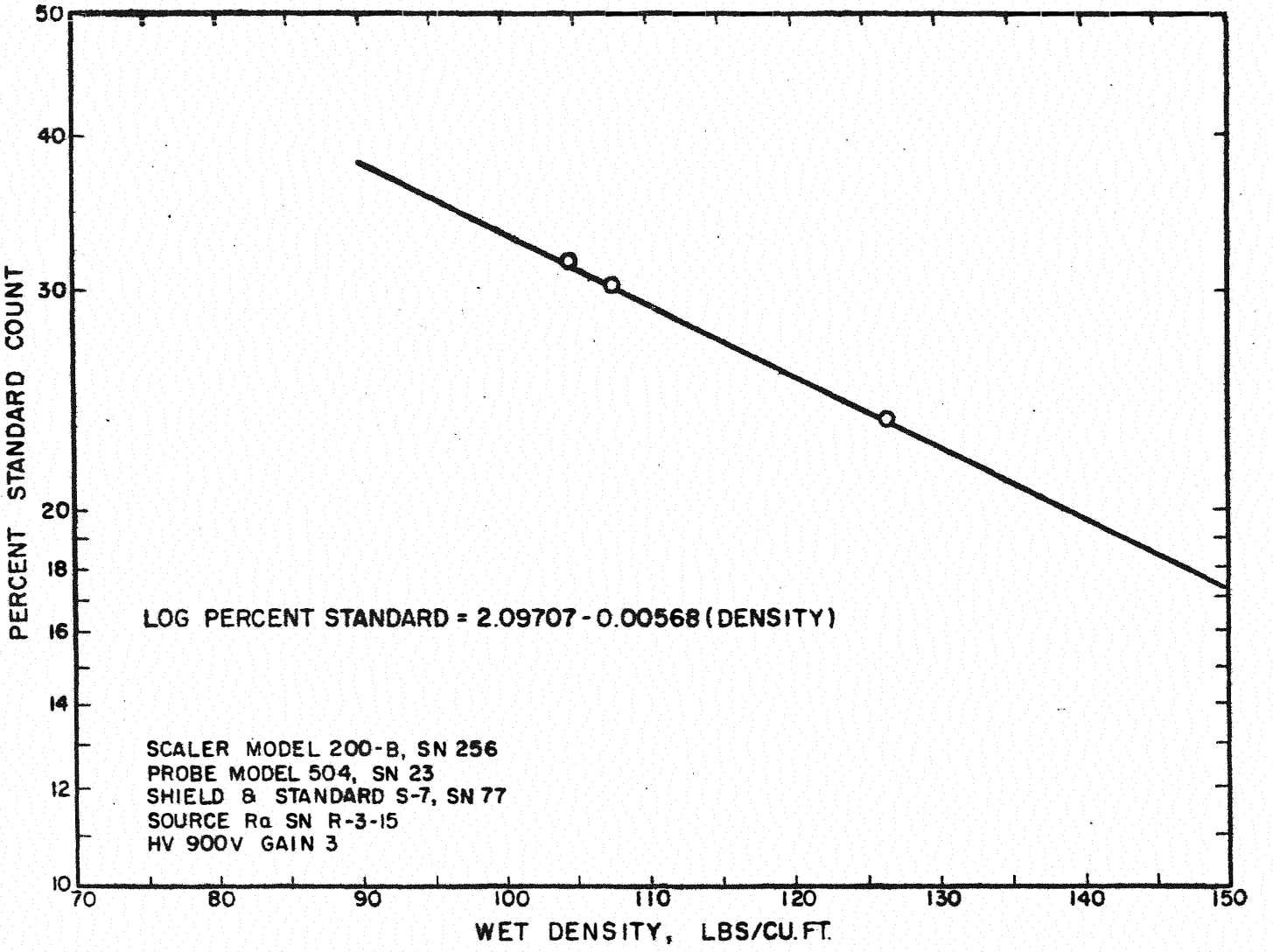

Fig 16. Density Calibration Curve-River Gravel

is exactly the same.

The slope of the data curve for the expanded shale standard shown in Fig 17 is quite different from that of the two aforementioned curves. The true or measured mass densities of this standard in the various conditions were much smaller than those of the other materials. For purposes of comparison the data were plotted and the curve extrapolated to the same wet density region of the other materials, as shown. Because the relationship may not be linear in the lower density range, below approximately 90 pcf, it is doubtful that this data extrapolation is valid. For this reason the expanded shale data was not used in the development of the recommended calibration curve.

Figure 18 shows the data plot for the crushed limestone barrel standard. The plotted points fit the linear theory very well and the curve equation is quite similar to those obtained for the limestone waste can standard and the river gravel.

The linear relationship for the Permian red clay standards is shown in Fig 19. The data point for the first barrel standard that was constructed is shown but there is some doubt as to its accuracy and it was not considered in the equation determination or in construction of the curve. A malfunction of the scaler unit became apparent at the time the readings were being made on this standard. This required the replacement of the unit driver tube and it is believed that this may have had something to do with the lack of fit of this data with that obtained from the two subsequently prepared clay standards.

While there is a certain amount of dissimilarity in the curve equation for the clay and those for the more granular

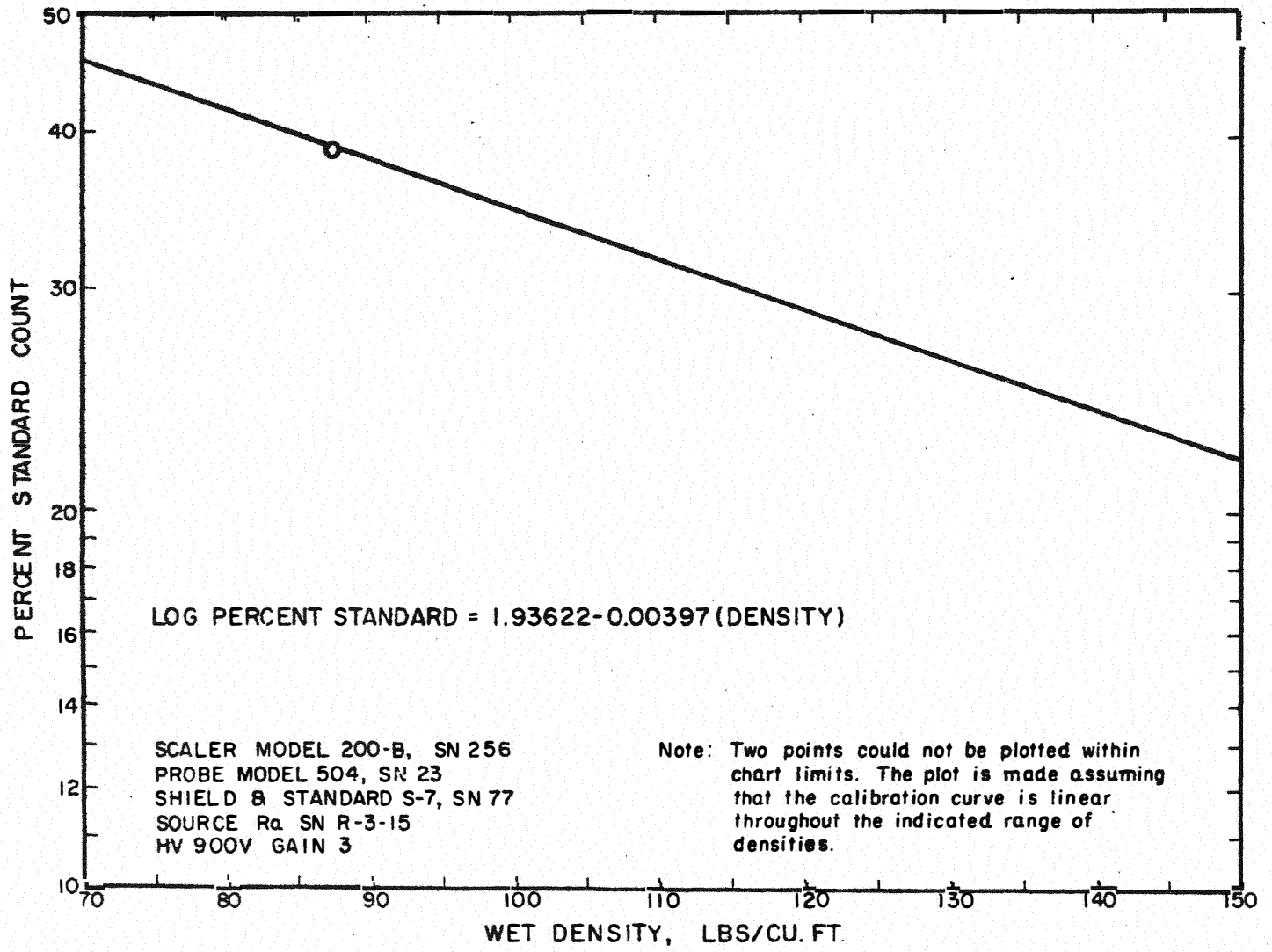

Fig 17. Density Calibration Curve-Expanded Shale

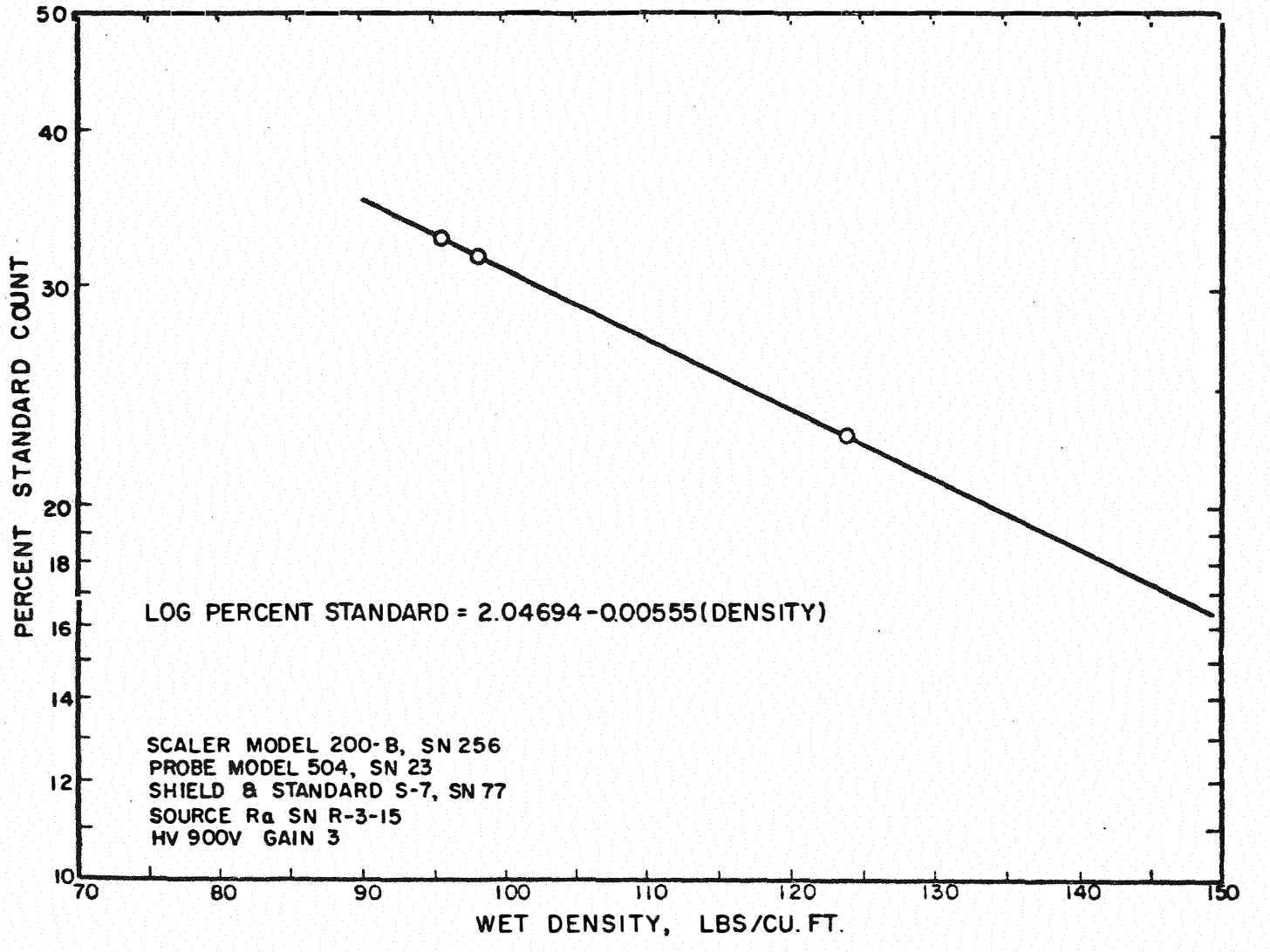

Fig 18. Density Calibration Curve-Limestone Barrel

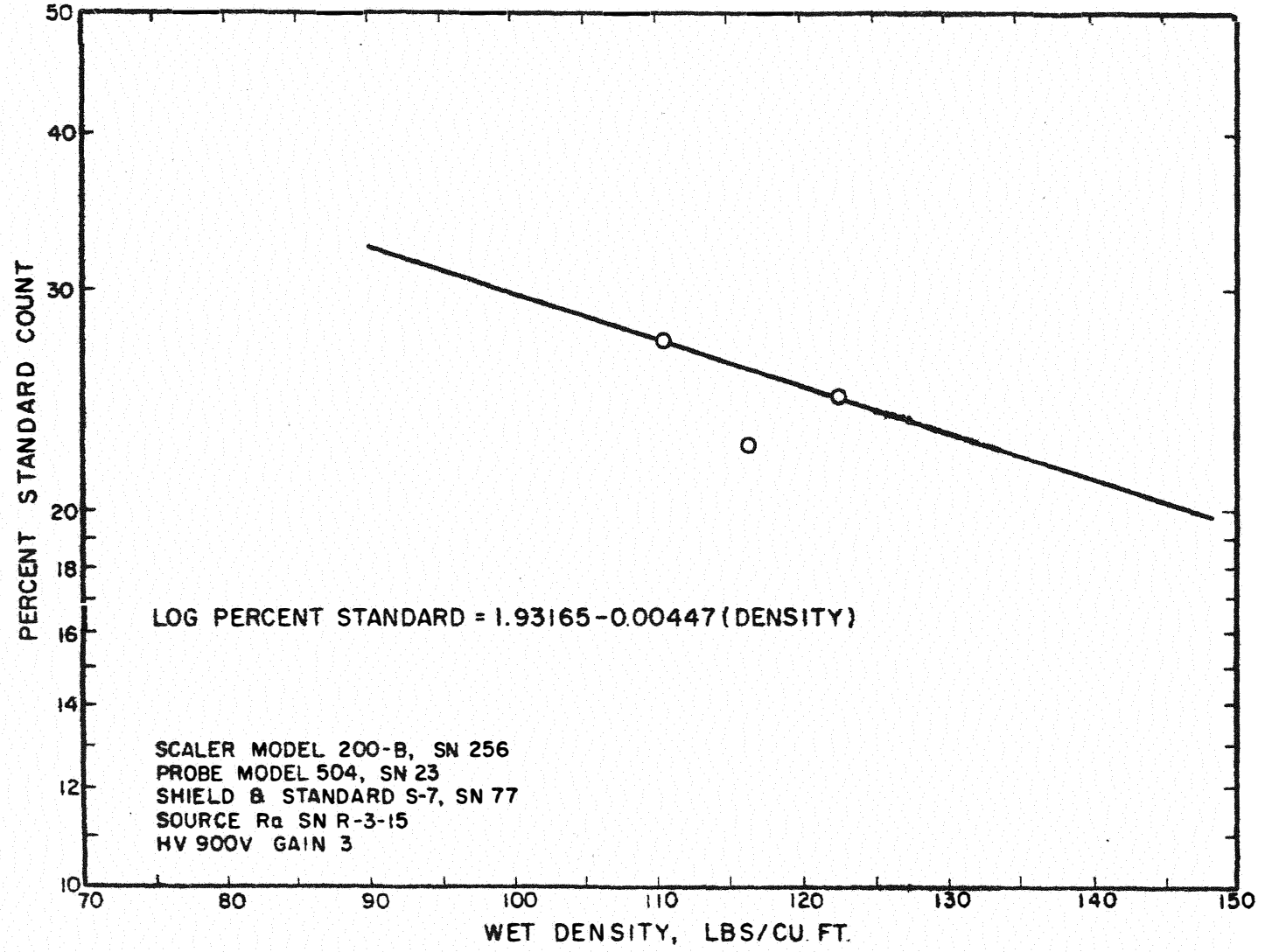

Fig 19. Density Calibration Curve-Permian Red Clay

materials, tests on additional clay standards may result in an increased slope of the clay curve and bring the respective curves more nearly parallel. The preparation and testing of additional permian clay standards as well as standards from other types of materials encountered during the drilling phase of the project is anticipated.

Figure 20 shows the combined data for all the standards tested. As indicated by the dashed lines, a rather narrow band of values is obtained from this combined plot. A single calibration curve and its equation is shown in Fig 21. This curve is the median of the band and for a given percent standard count the wet density of the material within $\pm$ 4 pcf can be determined from the curve. This range is the lateral spread, right and left of the median curve, within the calibration band.

At a percent standard count reading corresponding to a wet density of 90 pcf on the median calibration curve the error indicated by the lateral band width amounts to $\pm$ 4.4 percent. For lower percent standard count readings (higher density range) the percent error would be reduced. Using the median curve, the maximum error for the various materials tested occurred in the case of the crushed limestone barrel standard in the drained condition. The data point for this standard lies outside of the calibration band and the wet density determined from the curve is only 4.38 percent in error.

Since conventional methods of determining in place density of soils can be in error by as much as $\pm$ 10 percent (Ref 1) and

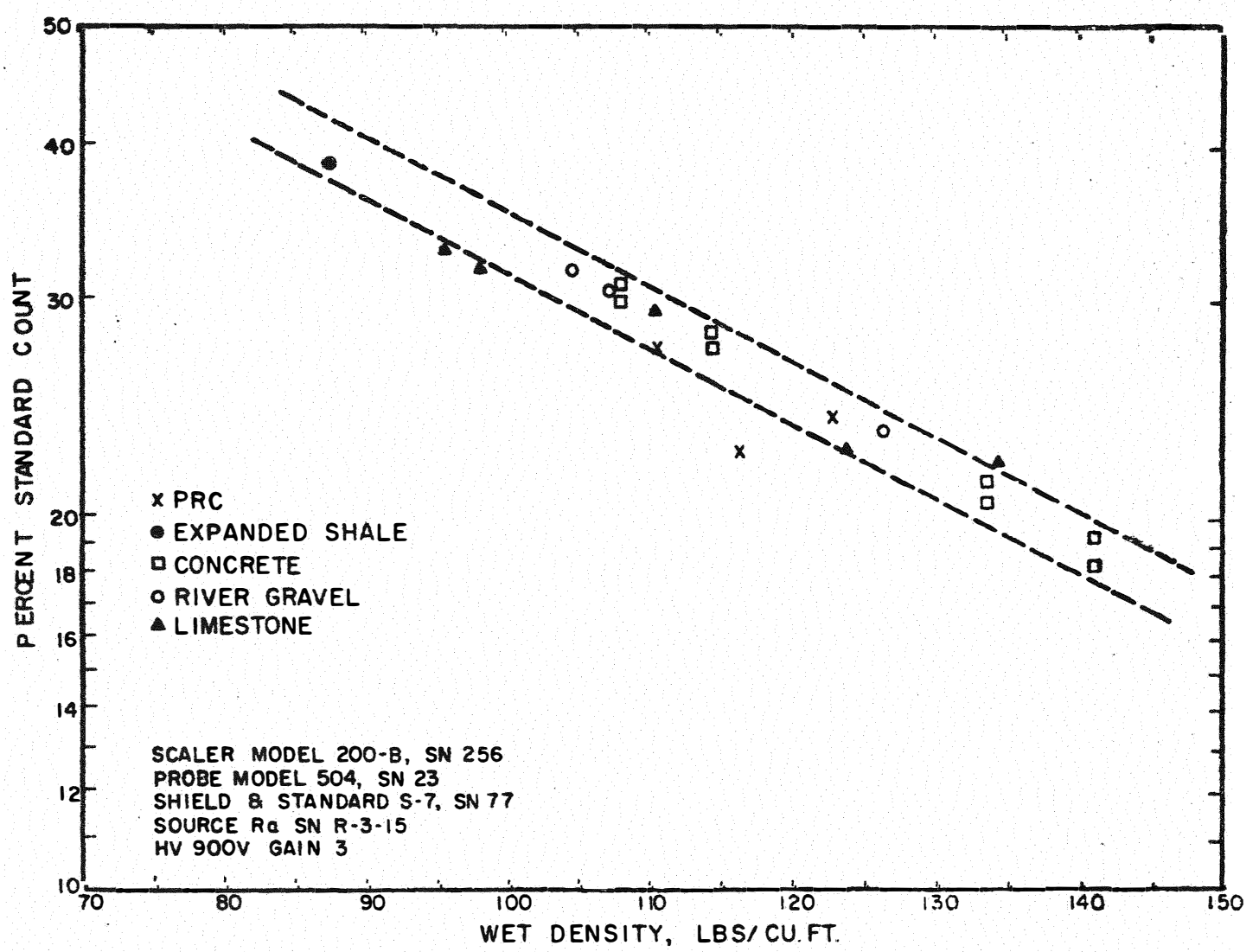

Fig 20. Combined Density Calibration Data

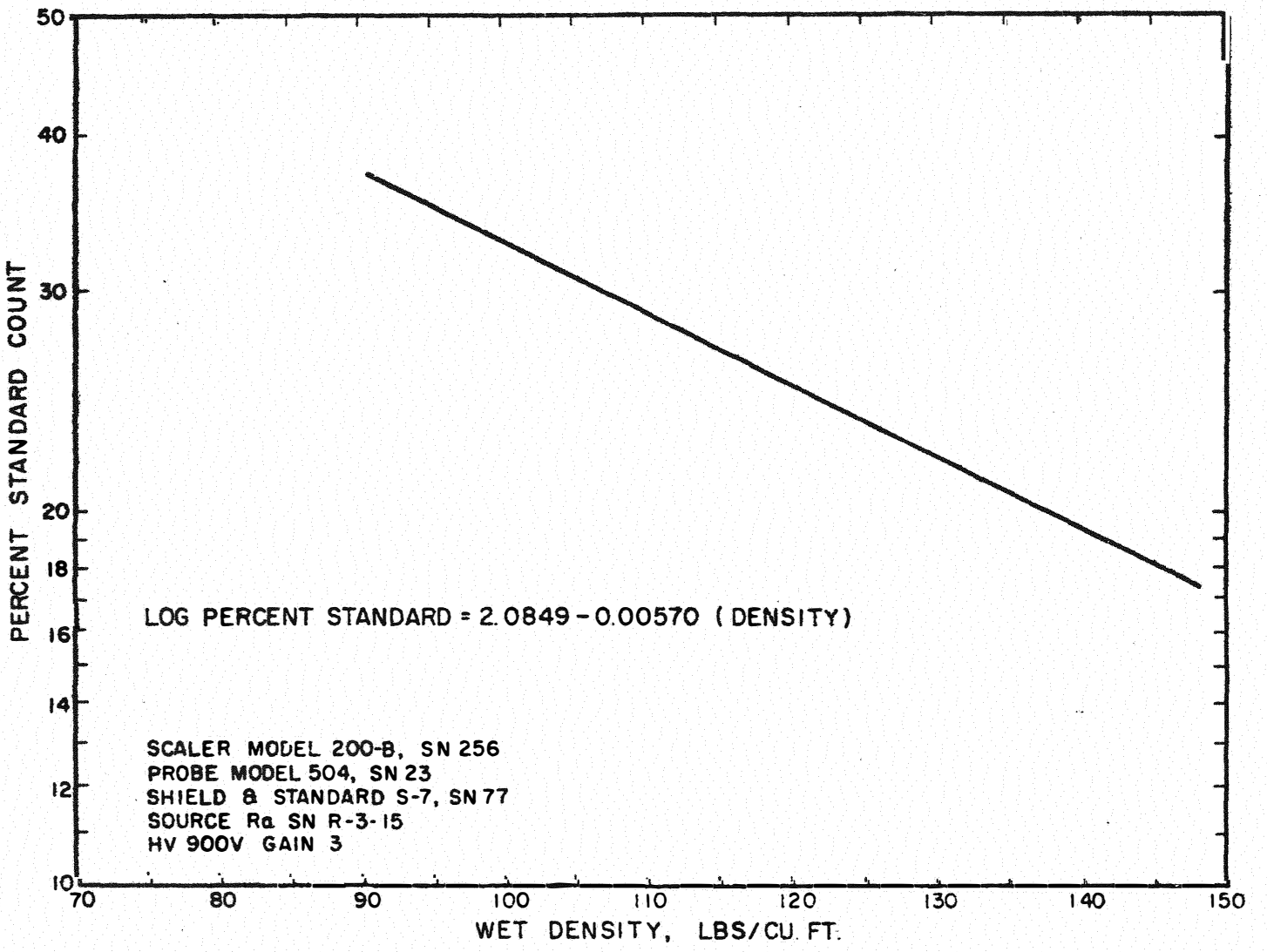

Fig 21. Recommended Median Calibration Curve

the maximum error experienced in this study was considerably less, it is believed that this band width type approach to the calibration of nuclear depth density equipment has considerable merit. Apparently mineral variances in soil do produce differences in nuclear response, and exacting calibration of these instruments may not be possible. This method, at least, indicates a practical solution to the calibration problem. However, the construction and testing of additional barrel standards utilizing a wide variety of Oklahoma soils will be necessary to validate this approach.

Certain problems in the construction of the barrel standards and sources of error in the measurements, particularly those composed of loose aggregates, are recognized. The density probe readings in the loose aggregate standards may possibly be in error due to an excess of pore space adjacent to the central access tube. This would also affect the moisture probe readings. Also the determination of the exact volume occupied by the aggregate mass in the barrel is subject to error due to difficulties in leveling the top surface. This could cause an error in the calculated density of the aggregate. In addition, the aggregates were vibrated into place in the dry state and when water was added to achieve the saturated condition slight settlements could have occurred. Settlement was noted in the expanded shale material and this required the complete reconstruction of the standard. These problems are simplified and a good bit of the error is eliminated if fine grained materials are used in the construction of the standards. However, if careful and painstaking construction procedures

are followed with the more granular materials good results can be achieved.

Moisture

The sphere of influence of a nuclear depth moisture probe is a function of the amount of water present. This is indicated by the following relationship:

$$R = 5.9 \left(\frac{100}{\text{Percent water by volume}} \right)^{1/3}$$

where R = radius of the sphere of influence in inches and percent water by volume = $\frac{\text{Pounds of water/cu ft x 100}}{62.4}$

As moisture content decreases the sphere of influence of the probe becomes greater. Thus, the moisture probe readings for the dry and drained conditions of the aggregate standards could not be relied upon due to the fact that the diameter of the sphere of influence at these low moisture contents was approaching or greater than that of the barrel standards. The readings for these conditions, as shown in Table 2, do indicate that there is little if any hydrogenous or thermalizing matter present in the aggregate materials.

A sample of the raw data sheet used to record the moisture probe readings is shown in Fig A-2 in the Appendix. This particular data sheet shows the readings made on the second Permian red clay standard. Notice that ten reference standard counts were taken prior and ten reference counts after the probe readings were made on the clay material. Simple averages of the standard reference and material counts are used to determine the percent of standard count. The same type of

data sheet and recording procedure was used for the depth density determinations.

Figure 22 shows the calibration curve for the moisture probe supplied by the manufacturer. The curve shown in Fig 23 was developed almost a year later by project personnel using the same instrument and cadmium chloride standards used by the manufacturer. Similar to the first two density calibration curves presented, these curves are essentially parallel but the latter one is displaced slightly upward from the first. This is also attributed to the use of a smaller number of reference standard counts in the second calibration sequence.

These curves are arithmetic plots with percent standard count as the ordinate and water content in pounds per cubic as the abscissa. The curves seem to be linear in the region from about 5 to 20 pounds of water per cubic foot of mass. At very low moisture contents the calibration curve is not accurate, since theoretically it should pass through the origin. A reading taken in pure water defines the upper limiting value and indicates that at high moisture contents the plot becomes curvilinear.

Figure 24 shows the combined data curve for the OSU laboratory standards. The saturated expanded shale and the doubtful Permian red clay points are shown but were not considered in drawing the curve. A good linear fit was obtained within the range of moisture contents tested.

In Fig 25 the linear portions of the two calibration curves on the Troxler cadmium chloride standards are contrasted with the one developed for the OSU laboratory standards.

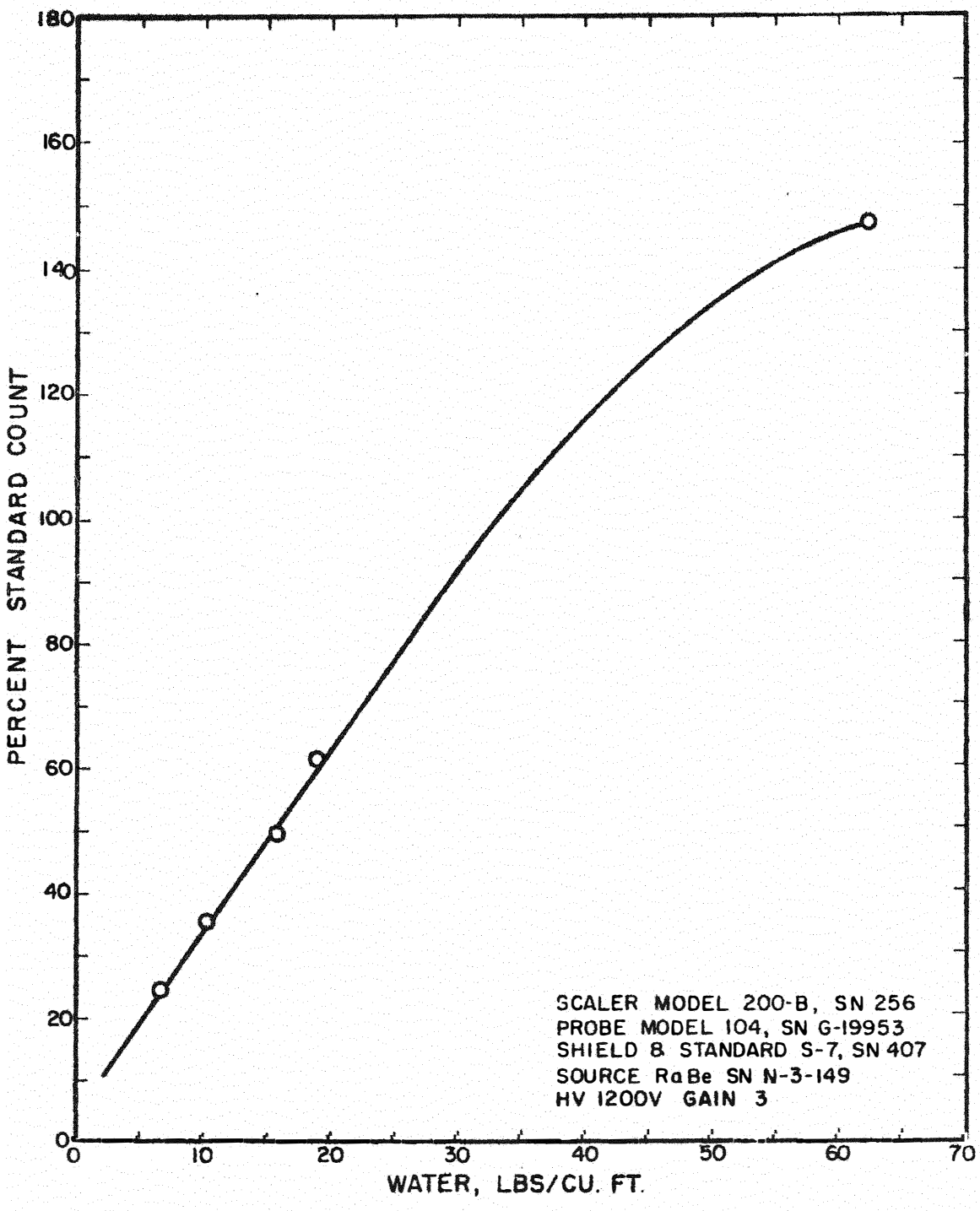

Fig 22. Moisture Calibration Curve-Troxler Cadmium Chloride, December 10, 1964

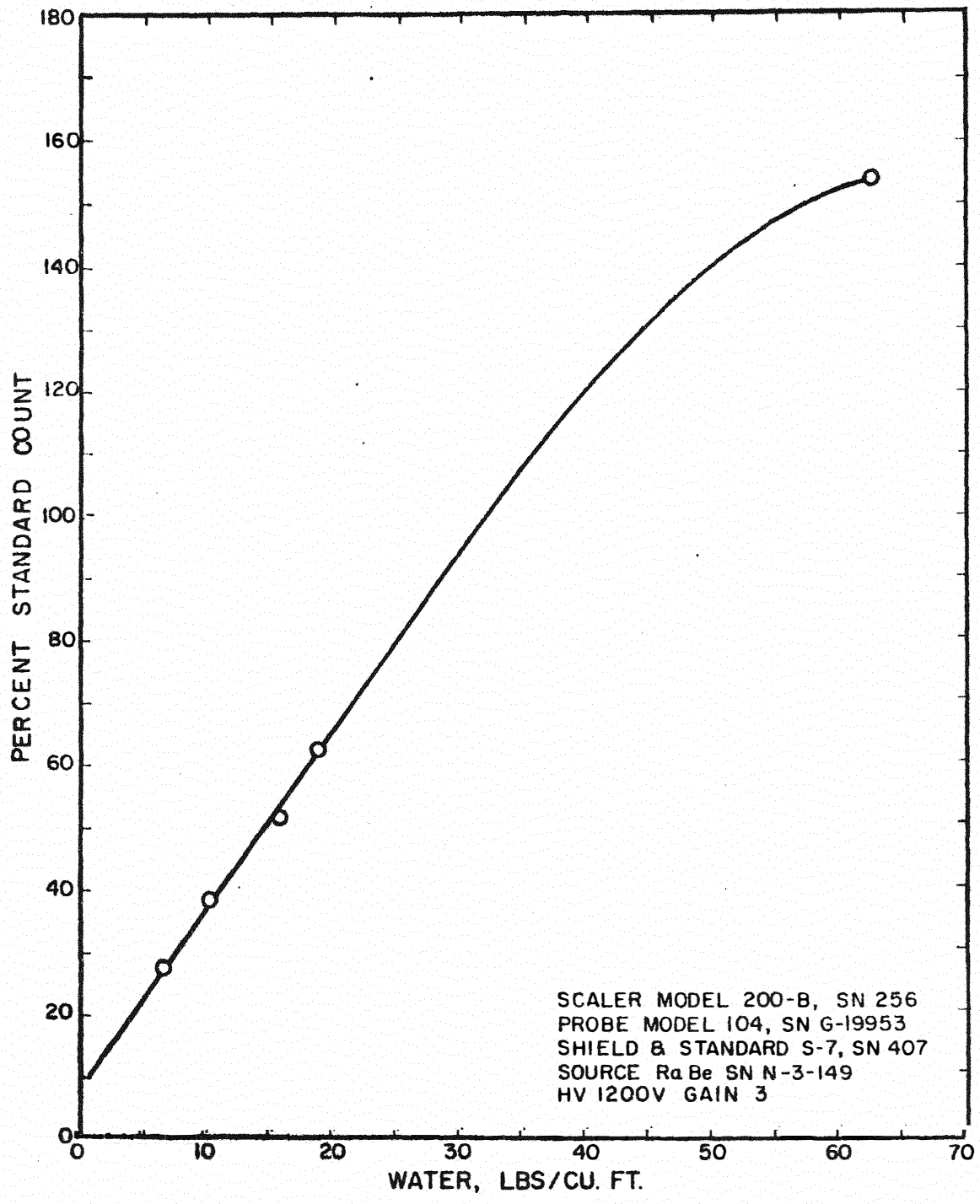

Fig 23. Moisture Calibration Curve-Troxler Cadmium Chloride, November 19, 1965

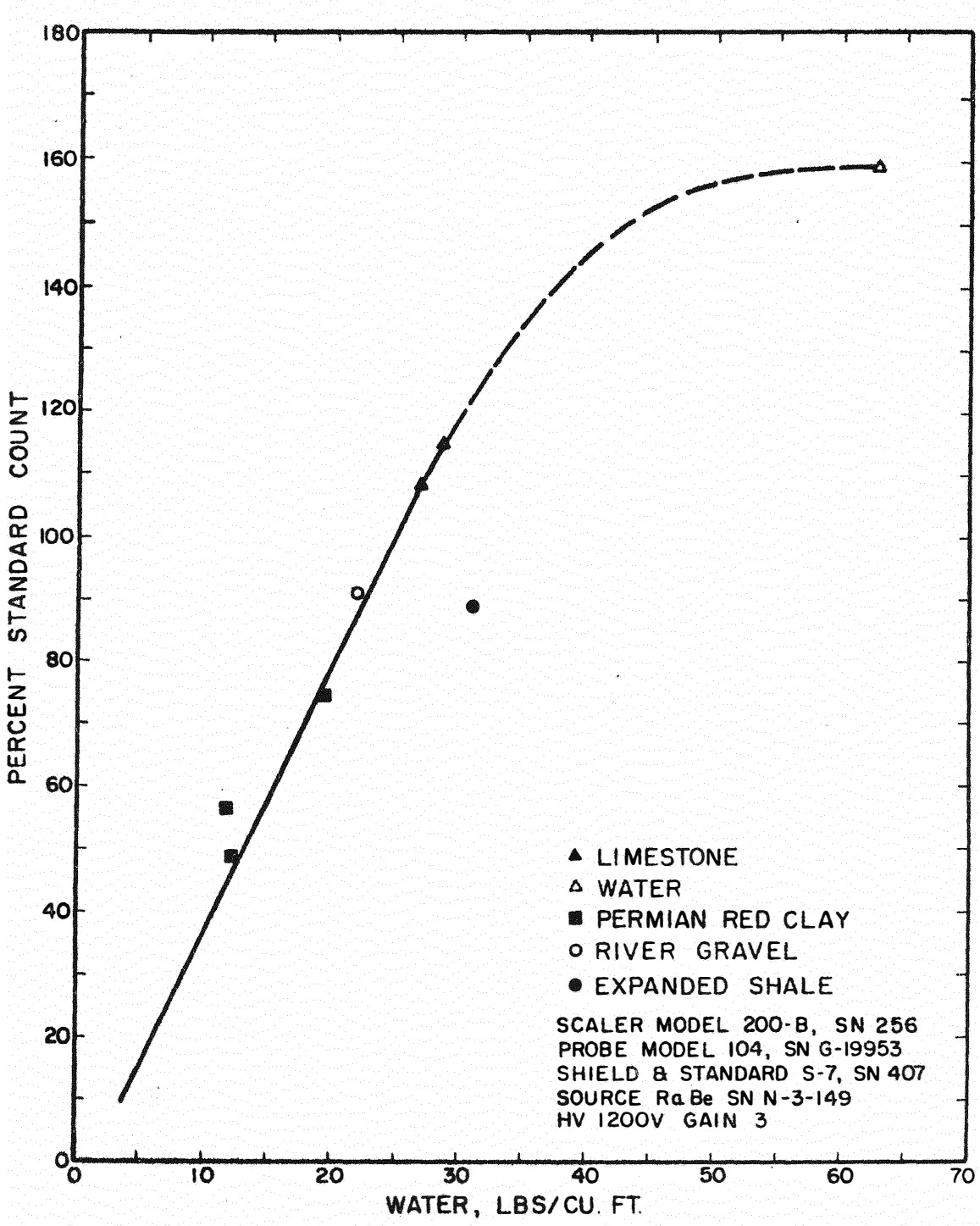

Fig 24. Moisture Calibration Curve-OSU Laboratory Standards

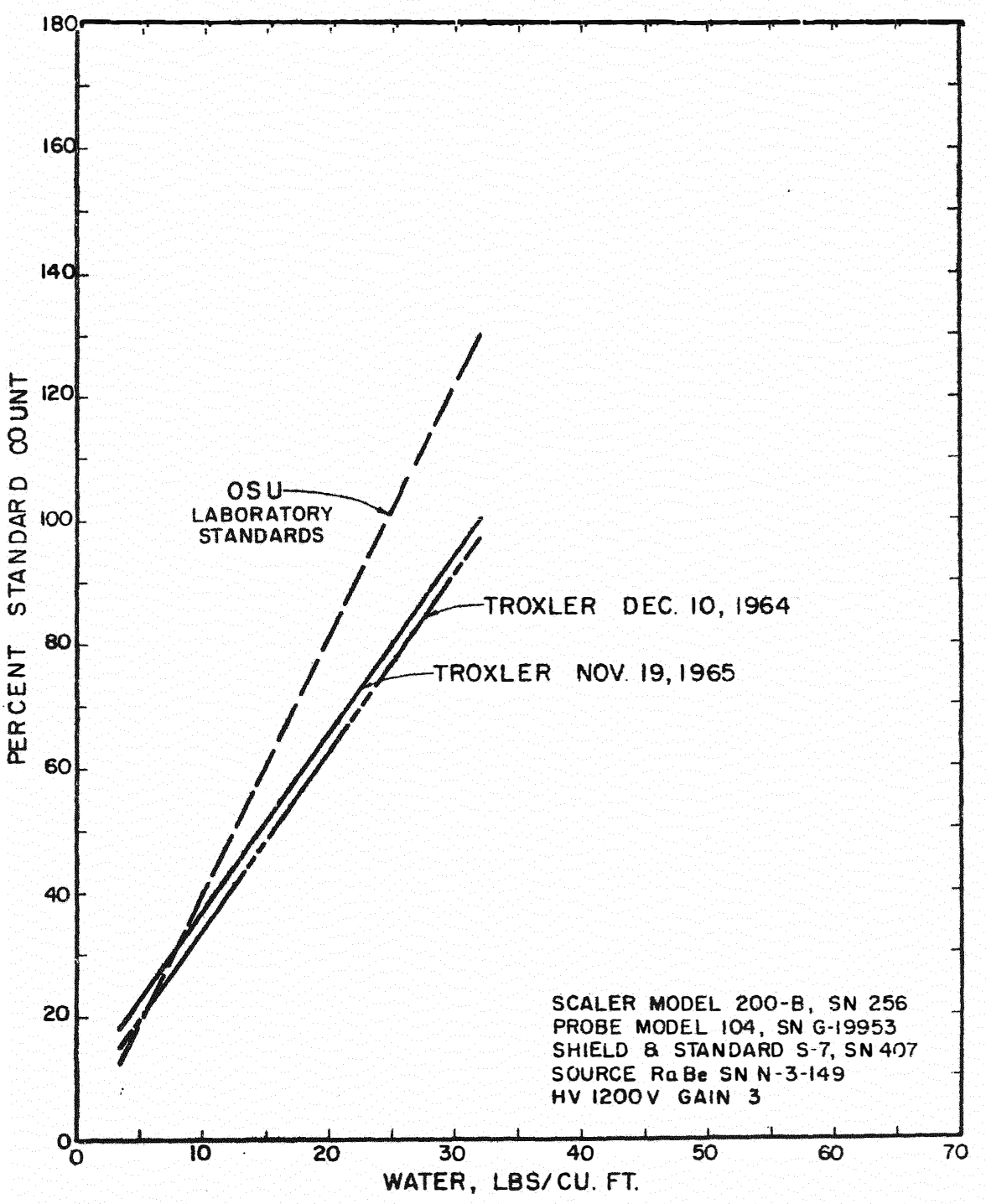

Fig 25. Moisture Calibration Curves-OSU and Manufacturer's Standards

The parallelism of the two Troxler curves is quite apparent here and a considerable slope change between the curves for the soil and non-soil type of standards is illustrated. This slope deviation in the two sets of curves indicates definite calibration differences for various standard media and the need for calibration of the moisture probe using materials similar to those that will be encountered during field usage.

Fig 26 shows the recommended moisture calibration curve for field use developed in this study. An equation for the linear portion of the curve between 5 and 30 pounds of water per cubic foot is indicated. Care should be exercised in employing this curve for percent standard counts less than 20 and greater than 120.

There is some question as to the exact linearity of these moisture probe calibration curves even in the lower ranges of moisture content. The development and measurement of additional soil type standards will help to resolve this question. If additional data should indicate a curvature in the region presently assumed to be linear it is believed that the change will be very slight and that the recommended curve can be used for field measurements with a reasonable degree of reliability.

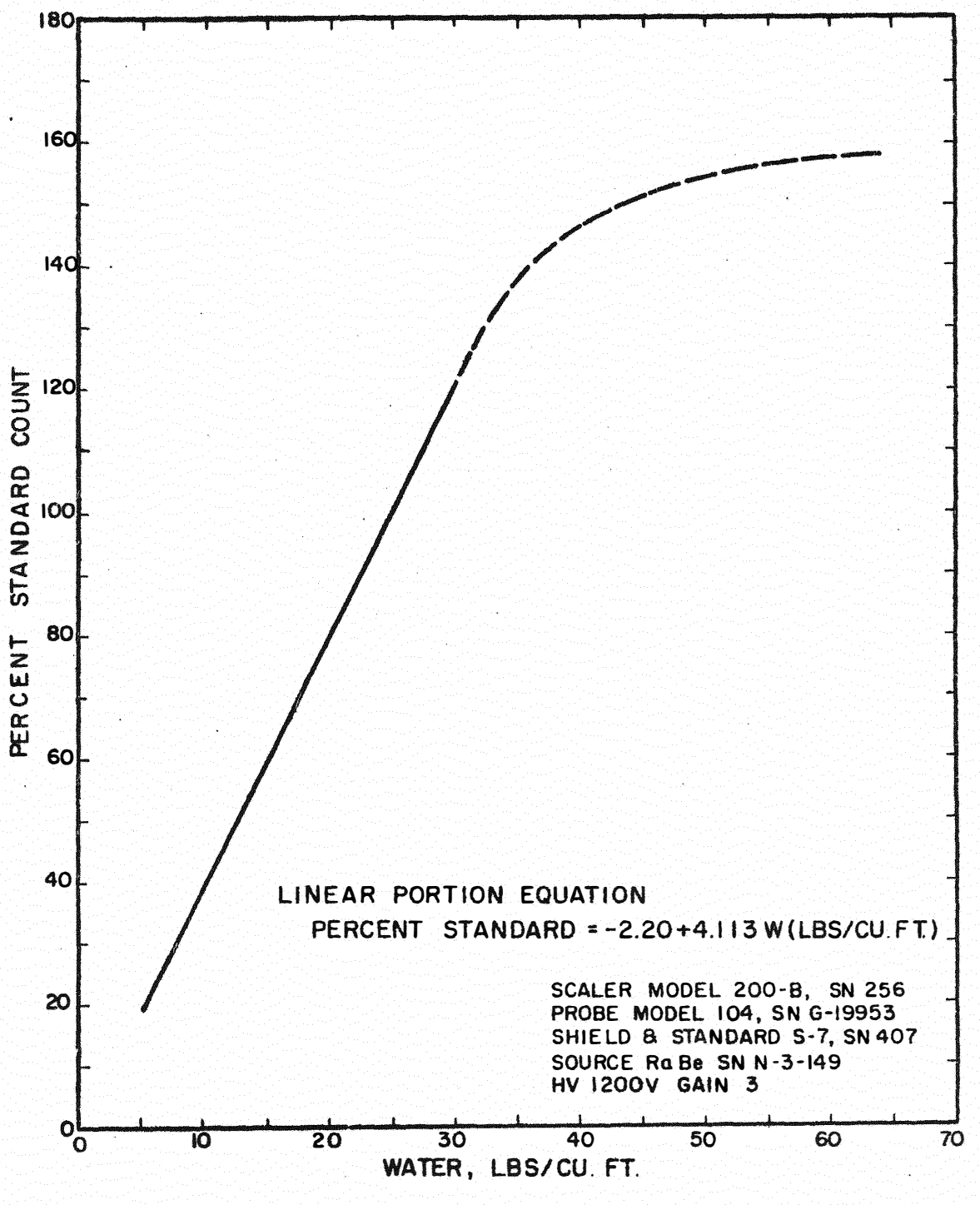

Fig 26. Recommended Moisture Calibration Curve

CHAPTER 5. SUMMARY AND CONCLUSIONS

The approach used in this study to construct secondary laboratory standards and develop a suitable calibration procedure for nuclear depth moisture/density probes has been successful in several respects. The following accomplishments are noted:

1. The handling and storage problems associated with extremely large laboratory standards have been minimized by utilizing cut-down 55-gallon oil drums as containers. The filled containers, although heavy, are easily handled using an overhead crane or a steel framework mounted on wheels with a chain sling type of suspension system.
2. Procedures for construction of the barrel standards utilizing both coarse and fine grained materials as calibration media have been developed.
3. Gravimetric measurement methods as well as the techniques of nuclear moisture/density determination have been worked out for the barrel standards.
4. Selected barrel standards can be saved and

preserved in their original moisture and density conditions for long periods of time. These will provide the means for rather precise "standardization" of the nuclear probes prior to field usage.

5. Although subject to certain construction problems and possible sources of error, the aggregate standards provided enough data for tentative calibration curves. The difficulties experienced with these standards are an additional argument for the use of finer grained materials more closely approximating actual subgrade soils as standard media.

6. The data obtained show a distinct change in nuclear response with a change in type of standard medium. This was evident in both the density and moisture calibrations and indicates a definite need for calibration of the respective probes using materials similar to those that will be encountered in the field.

7. As previously mentioned, there is considerable variance in response of the moisture probe between soil and non-soil type of standard media. The results obtained with the aggregate and clay soil barrel standards indicate a linearity of the calibration curve between 5 and 30 pounds of water per cubic foot and the developed curve should yield reliable

results from field measurements.

8. While the density results indicate that an exacting calibration, i.e, one that will cover or apply to all soil types encountered, will not be possible, the variance does not appear extremely great. The "band width" approach to calibration of the density probes offers a practical solution to this problem with a minimum amount of error involved over the normal range of soil densities. At the present time the width of this density calibration band may be too wide to detect minor differences in absolute values but it is quite capable of detecting major discrepancies and certainly even minor variations or changes with time.

The median curve developed from the band of density calibration values is recommended for use in field density determinations. For ease in field use this median curve can be converted to tabular form to eliminate variations in curve interpretation by field personnel. Since the primary use of density values in the Subgrade Moisture Variations project is to allow computation of moisture content on a dry weight basis, density checks on highway subgrade soils can probably be made on a yearly or semi-annual basis. There should be little need to make density checks on the same time schedule as the more frequent moisture observations unless fluctuations in moisture readings at a particular location indicate large or progressive volume changes in the subgrade material.

At present, it is not planned to obtain undisturbed samples for density determinations at the time of installation of the access tubes beneath highway pavements. However, it is anticipated that disturbed soil samples from various depths can be easily obtained during the drilling phase of the tube installation and the original field moisture conditions determined from these samples. These field moisture contents can be used to correlate the laboratory and field obtained nuclear moisture data with the actual conditions that exist in the field.

There is a definite need for a more detailed investigation of the variance in nuclear response of both the moisture and density probe with change in soil type. This is necessary to determine the extent of this variance on Oklahoma soil types and the further validate the "band width" type of approach to calibration. In this regard, it is recommended that this study be extended and that additional barrel standards using a variety of Oklahoma soil types as calibration media be constructed and evaluated.

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APPENDIX

SAMPLE DATA SHEETS

LEAST SQUARES REGRESSION LINE ANALYSIS

FORM FOR CALCULATION AND PRESENTATION OF RESULTS Date: April 12, 1966

Given: Permian Red Clay - Sample #2

True Mass Density X	Probe Reading % std.	Log. Probe Reading Y		
122.7	24.18	1.38346		
110.3	27.47	1.43886		

n = 2

ΣX = 233.0 ΣY = 2.82232

$\bar{X}$ = 116.5 $\bar{Y}$ = 1.41116

ΣX^2 = 27221.38 ΣY^2 = _____ ΣXY = 328.45680

$(\Sigma X)^2/n$ = 27144.50 $(\Sigma Y)^2/n$ = _____ $(\Sigma X)(\Sigma Y)/n$ = 328.80028

Σx^2 = 76.88 Σy^2 = _____ Σxy = -0.34348

$b_1 = \Sigma xy / \Sigma x^2$ = $-4.4677 (10^{-3})$

$b_o = \bar{Y} - b_1 \bar{X}$ = 1.93165

$\hat{Y} = b_o + b_1 X$ = 1.93165 - 0.0044677

Log Percent Standard = 1.93165 - 0.0044677 (Density)

Fig A-1. Sample Computation Sheet-Least Squares Regression Line Analysis

STANDARD REFERENCE READINGS

Scaler 200B SN 256
Probe Type: (Moisture); ~~Density~~
Source: (Ra 226 Be); ~~Ra 226~~
Reading Time: Long

Sample Type: Permian Red Clay
Sample No.: 2
Mass Density: 122.2 #/ft³
Moisture Content: 19.3 #/ft³

Date	29 Mar 66				
Gain Setting	3				
Voltage	1250				
Taken by	LeFevre	Marks	LeFevre	Marks	
1	11 657	8 574	8 700	11 687	
2	11 655	8 639	8 632	11 702	
3	11 730	8 709	8 667	11 867	
4	11 674	8 643	8 744	11 976	
5	11 801	8 830	8 711	11 656	
6	11 785	8 809	8 958	11 689	
7	11 492	8 674	8 783	11 998	
8	11 756	8 649	8 743	11 749	
9	11 758	8 855	8 717	11 944	
10	11 789	8 876	8 698	11 812	
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
Total	117097	87276	87353	116080	
Average	11710	8728	8735	11608	
Comments	Shield	Std. barrel @ 7½" fr. bottom	Std. barrel	Shield	

Calculations: Shield 11710 + 11808 = 23,518 Barrel 8728 + 8735 = 17463

$\frac{17,463}{23,518} \times 100 = 74.25\%$ (Avg. Percent Std.)

Fig A-2. Sample Data Sheet